Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Stable Analogues of Nojirimycin – Synthesis and Biological Evaluation of Nojiristegine and *manno*-Nojiristegine

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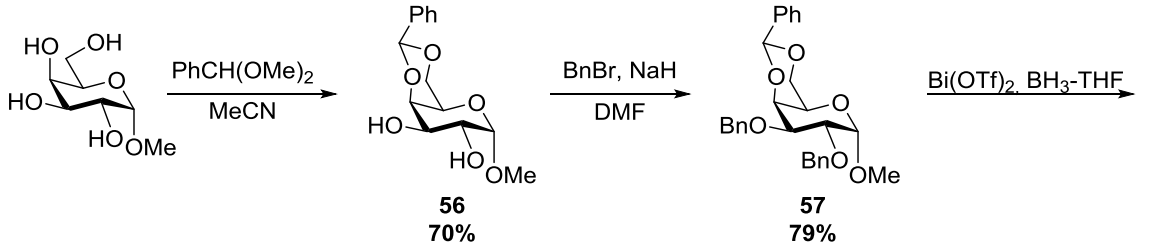
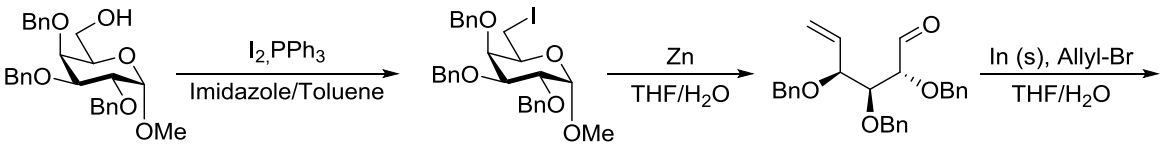


56
70%

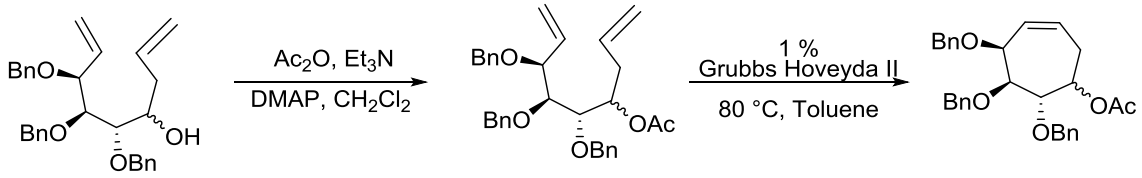
57
79%



58
83%

59
73%

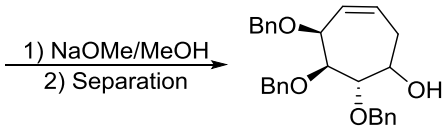
60



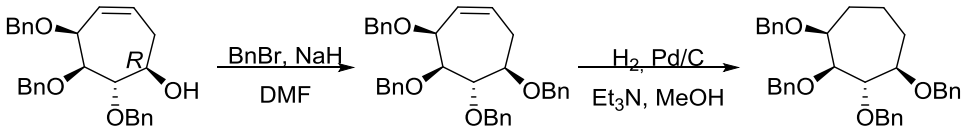
61
R/S 1:2.1

62
59% over 3 steps

63
83 %



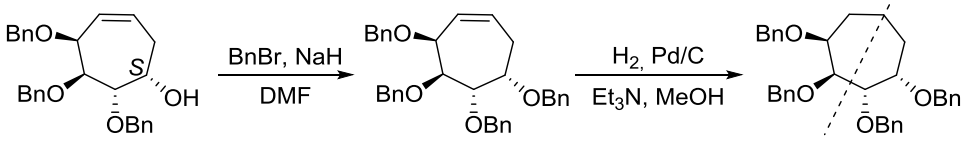
64 and **65**
90%



64

66
72%

68
40%

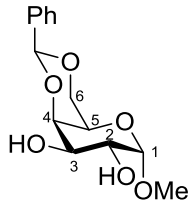


65

67
71%

69
79%
(symmetrical)

Methyl 4,6-*O*-Benzylidene-α- D-galactopyranoside (56):



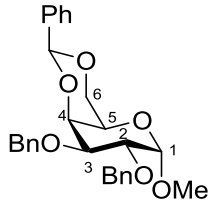
56

Methyl α- D-galactopyranoside (5.00 g, 25.8 mmol) was dissolved in anhydrous chloroform (362 mL) and stirred under a nitrogen atmosphere in a distillation setup. Benzaldehyde dimethyl acetal (5.48 mL, 36.4 mmol, 1.4 eq.) and 10-camphorsulfonic acid monohydrate (93.8 mg, 0.40 mmol, 0.02 eq.) were added, and the mixture heated to reflux upon which approximately 150 mL chloroform/methanol was removed by distillation (1 atm.) during the reaction time. After 7 h the reaction was allowed to cool to rt. and quenched by adding triethylamine (2.50 mL) and water (125 mL). The organic phase was dried over $MgSO_4$, filtered and concentrated under reduced pressure. The resulting white powder was washed with pentane (50 mL) and EtOAc (50 mL), and dried overnight on a vacuum line, yielding pure methyl 4,6-*O*-benzylidene-α- D-galactopyranoside (**56**) as a white solid (70%, 5.06 g).

R_f (ethyl acetate/ethanol) 0.43; M_p(Corr.) 163.4-164.6 °C (±2.6 °C); $[\alpha]_D^{295K}$ +129 (c 0.9, $CHCl_3$); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.53-7.46 (m, 2H, Ar*H*), 7.41-7.34 (m, 3H, Ar*H*), 5.56 (s, 1H, PhC*H*), 4.94 (d, 1H, $J_{1,2}$ 3.1 Hz, H1), 4.34-4.25 (m, 2H, $H6_{eq}$, H4), 4.09 (dd, 1H, J_{gem} 12.6 Hz, $J_{5,6ax}$ 1.9 Hz, $H6_{ax}$), 3.95-3.87 (m, 2H, H2, H3), 3.73-3.70 (m, 1H, H5), 3.47 (s, 3H, OCH_3), 2.36 (bs, 1H, OH), 2.09 (bs, 1H, O*H*). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C 138.1 (Ar*C*), 129.7 (*p*Ar*a*-Ar*C*), 128.7 (2x *orto*-Ar*C*), 126.7 (2x *meta*-Ar*C*), 101.6 (*C*HPh), 100.5 (C1), 76.0 (C4), 70.0 (C2/3), 69.9 (C2/3), 69.4 (C6), 62.8 (C5), 55.7 (O*C*H_3). IR: ν_{max} (cm^{-1}): 3469 (w, OH), 2906 (w, sp^3 carbons), 1045 (s), 1028 (s, acetal), 741 (s, aromatic carbon), 695 (s, aromatic carbon). HRMS(ESI): calcd. for $C_{14}H_{18}O_6Na$: 305.1001; found 305.0998,

NMR data was in accordance with preciously reported values.[2,3]

Methyl 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-α- D-galactopyranoside (57):



57

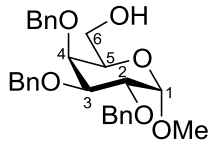
Methyl 4,6-*O*-benzylidene-α- D-galactopyranoside (**56**) (4.35 g, 15.4 mmol) was dissolved in anhydrous DMF (60.0 mL) and stirred under a nitrogen atmosphere. The mixture was cooled to 0 °C and added 60% NaH in mineral oil (1.16 g, 29.0 mmol, 1.9 eq.) and stirred for 10 min. before BnBr (4.46 mL, 31.3 mmol, 2.4 eq.) was added and the mixture allowed to slowly reach rt. over 6 h. Additional 60% NaH in mineral oil (0.60 g, 15.0 mmol, 1.0 eq.) and BnBr (2.00 mL, 16.8 mmol, 1.1 eq.) was added and the mixture stirred overnight. The reaction was quenched by slow addition to a large Erlenmeyer flask containing ice water, upon which the product precipitated. The mixture was filtered and dried overnight on a vacuum line, yielding pure methyl 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-α- D-galactopyranoside (**57**) as a white solid (79%, 5.61 g).

R_f (pentane/ethyl acetate 7:3) 0.22; M_p(Corr.) 167.9-171.3 °C (±2.6 °C); $[\alpha]_D^{295K}$ +75.0 (c 1.1, $CHCl_3$); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.57-7.26 (m, 15H, Ar*H*), 5.48 (s, 1H, C*H*Ph), 4.88 and 4.68 (d, each 1H, $J_{A,B}$

12.0 Hz, CH_AH_BPh), 4.84 and 4.74 (d, each 1H, $J_{A,B}$ 12.3 Hz, CH_AH_BPh), 4.21 (dd, 1H, J_{gem} 8.2 Hz, $J_{5,6eq}$ 3.4 Hz, H6$_{eq}$), 4.77 (d, 1H, $J_{1,2}$ 3.5 Hz, H1), 4.18 (dd, 1H, $J_{3,4}$ 3.4 Hz, $J_{4,5}$ 2.6 Hz, H4), 4.07 (dd, 1H, $J_{2,3}$ 10.1 Hz, $J_{3,4}$ 3.4 Hz, H3), 4.01 (dd, 1H, J_{gem} 8.2 Hz, $J_{5,6ax}$ 1.8 Hz, H6$_{ax}$), 3.98 (dd, 1H, $J_{2,3}$ 10.1 Hz, $J_{1,2}$ 3.5 Hz, H2), 3.59 (bs, 1H, H5), 3.39 (s, 3H, OCH$_3$). ^{13}C-NMR (CDCl$_3$, 100 MHz): δ_C 138.8 (Ar*C*), 138.6 (Ar*C*), 137.9 (Ar*C*), 128.9, 128.3, 128.1, 128.1, 127.7, 127.6, 127.6, 126.4 (All Ar*C*), 101.1 (*C*HPh), 99.5 (C1), 77.4 (C2), 75.5 (C3), 74.8 (C4), 73.8 (*C*H$_2$Ph), 72.2 (*C*H$_2$Ph), 69.4 (C6), 62.5 (C5), 55.5 (O*C*H$_3$). IR: ν_{max} (cm^{-1}): 3029 (w, sp^2 carbons), 2981 (w, sp^3 carbons), 1050 (s, ether/acetal), 1028 (s), 1017 (s), 737 (s), 708 (s), 695 (s, aromatic). HRMS (ESI): calcd. for $C_{28}H_{30}O_6Na$: 485.1940; found 485.1936.

NMR data was in accordance with preciously reported values.[3]

Methyl 2,3,4-tri-*O*-benzyl-6-hydroxy-α- D-galactopyranoside (58):



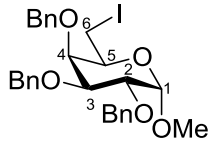
58

BH$_3$:THF complex in THF (1 M, 46.0 mL, 46.0 mmol, 2.0 eq.) was added to methyl 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-α-D-galactopyranoside (**57**) (10.6 g, 22.9 mmol) in anhydrous dichloromethane (200 mL) and stirred under inert atmosphere at r.t.. The mixture was stirred for 10 min. before addition of Bi(OTf)$_3$ (2.01 g, 1.15 mmol, 0.05 eq.). The reaction was quenched after 20 min. by slow addition to a large Erlenmeyer flask containing sat. NaHCO$_3$. The water phase was extracted with 3x dichloromethane, and the combined organic phases dried over MgSO$_4$, filtered and concentrated under reduced pressure. The crude compound was purified by flash column chromatography (pentane/ethyl acetate 1:1), yielding methyl 2,3,4-tri-*O*-benzyl-6-hydroxy-α- D-galactopyranoside (**58**) as a clear oil (83%, 8.84 g).

R_f (pentane/ethyl acetate 2:3) 0.45; $[\alpha]_D^{295K}$ +2.10 (c 1.0, CHCl$_3$); ^{1}H-NMR (CDCl$_3$, 400 MHz): δ_H 7.43-7.27 (m, 15H, Ar*H*), 4.98 and 4.64 (d, each 1H, $J_{A,B}$ 11.6 Hz, CH_AH_BPh), 4.90 and 4.76 (d, each 1H, $J_{A,B}$ 11.8 Hz, CH_AH_BPh), 4.85 and 4.70 (d, each 1H, $J_{A,B}$ 12.0 Hz, CH_AH_BPh), 4.71 (d, 1H, $J_{1,2}$ 3.7 Hz, H1), 4.06 (dd, 1H, $J_{2,3}$ 10.1 Hz, $J_{1,2}$ 3.7 Hz, H2), 3.95 (dd, 1H, $J_{2,3}$ 10.1 Hz, $J_{3,4}$ 2.9 Hz, H3), 3.88 (bd, 1H, $J_{3,4}$ 2.9 Hz, H4), 3.76-3.67 (m, 2H, H6$_{eq}$, H5), 3.52-3.44 (m, 1H, H6$_{ax}$), 3.37 (s, 3H, O*C*H$_3$). ^{13}C-NMR (CDCl$_3$, 100 MHz): δ_C 138.7 (Ar*C*), 138.4 (Ar*C*), 138.2 (Ar*C*), 128.6, 128.5, 128.4, 128.3, 128.1, 127.9, 127.7, 127.6, 127.5 (Ar*C*), 98.8 (C1), 79.1 (C3), 76.5 (C2), 75.1 (C4), 74.4 (*C*H$_2$Ph), 73.6 (*C*H$_2$Ph), 73.6 (*C*H$_2$Ph), 70.3 (C5), 62.4 (C6), 55.4 (O*C*H$_3$). IR: ν_{max} (cm^{-1}): 3472 (w, OH), 3030 (w, sp^2 carbons), 2901 (w, sp^3 carbons), 1044 (s, ether/acetal), 1027 (s), 734 (s), 695 (s, aromat). HRMS (ESI): calcd. for $C_{28}H_{32}O_6Na$: 487.2097; found 487.2091.

NMR data was in accordance with preciously reported values.[3]

Methyl 2,3,4-tri-*O*-benzyl-6-deoxy-6-iodo-α- D-galactopyranoside (59):



59

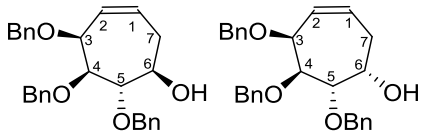
Imidazole (6.86 g, 100 mmol, 5.7 eq.), triphenyl phosphine (12.6 g, 48.6 mmol, 2.7 eq.) and iodine (11.3 g, 44.4 mmol, 2.5 eq.) was added to a solution of methyl 2,3,4-tri-*O*-benzyl-6-hydroxy-α-D-galactopyranoside (**58**) (8.25 g, 17.8 mmol) in anhydrous acetonitrile (17.6 mL) and anhydrous toluene (33.1 mL) under inert

atmosphere. The mixture was heated and stirred at 90 °C overnight, then quenched by cooling to rt. and adding solid $Na_2S_2O_3$ before diluting with dichloromethane. The organic phase was washed with water and brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography (pentane/ethyl acetate 9:1), yielding pure methyl 2,3,4-tri-*O*-benzyl-6-deoxy-6-iodo-α-D-galactopyranoside (**59**) as a transparent oil which turned slightly yellow over time (73%, 7.43 g).

R_f (pentane/ethyl acetate 19:1) 0.23; $[\alpha]_D^{295K}$ +23.0 (c 1.0, $CHCl_3$); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.38-7.18 (m, 15H, Ar*H*), 4.99and 4.58(d, each 1H, $J_{A,B}$ 11.3 Hz, C$H_A$$H_B$Ph), 4.85 and 4.70 (d, each 1H, $J_{A,B}$ 11.8 Hz, C$H_A$$H_B$Ph), 4.78 and 4.63 (d, each 1H, $J_{A,B}$ 12.1 Hz, C$H_A$$H_B$Ph), 4.59 (d, 1H, $J_{1,2}$ 2.4 Hz, H1), 3.97 (m, 2H, H2, H4), 3.88 (dd, $J_{2,3}$ 10.3 Hz, $J_{3,4}$ 2.8 Hz, H3), 3.79 (m, 1H, H5), 3.36 (s, 3H, OCH_3), 3.17 (dd, 1H, J_{gem} 10.1 Hz, $J_{5,6eq}$ 7.7 Hz, H6$_{eq}$), 3.02 (dd, 1H, J_{gem} 10.1 Hz, $J_{5,6ax}$ 6.2 Hz, H6$_{eq}$). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C 138.8, 138.5, 138.4, 128.5, 128.5, 128.5, 128.2, 127.9, 127.9, 127.7, 127.6, 99.0, 79.2, 76.1, 75.9, 75.1, 73.7, 71.4, 55.9, 3.6. IR: ν_{max} (cm^{-1}): 3029 (w, sp^2 carbons), 2902 (w, sp^3 carbons), 1092 (s), 1039 (s, ether/acetal), 1026 (s), 733 (s), 695 (s, aromat), 565 (w, C-I). HRMS (ESI): calcd. for $C_{28}H_{31}IO_5Na$: 597.1114; found 597.1108.

NMR data was in accordance with preciously reported values.[4]

(3*S*,4*S*, 5*S*, 6*R*)-3,4,5-Tri-*O*-benzyl-6-hydroxyl-cycloheptene (64) and (3*S*,4*S*, 5*S*, 6*S*)-3,4,5-Tri-*O*-benzyl-6-hydroxy-cycloheptene (65):



64 **65**

Pre-activated Zn dust[a] (7.72 g, 117 mmol, 13 eq.) was added to methyl 2,3,4-tri-*O*-benzyl-6-deoxy-6-iodo-α-D-galactopyranoside (**59**) (5.11 g, 8.90 mmol, 1.00 eq.) in THF (37.4 mL) and water (26.3 mL). The mixture was sonicated at 40 °C. After both 5 h and 7 h additional pre-activated Zn dust (3.71 g, 56 mmol, 6 eq.) was added. The mixture was sonicated at 40 °C, until completion was observed by ^{1}H-NMR analysis. Upon completion the reaction was cooled to rt., diluted with Et_2O and filtered through a Celite® plug. The organic phase was washed with water and brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure.

Indium (7.63 g, 67 mmol, 7.3 eq.) and allyl bromide (1.54 mL, 18 mmol, 2.0 eq.) were added to the crude aldehyde (**60**) in a solution of 50 V/V% THF/water (44 mL). The mixture was sonicated at 40 °C and additional portions of allyl bromide (1.54 mL, 18 mmol, 2.0 eq.) were added until full consumption of the starting material was observed by ^{1}H-NMR analysis. The mixture was then cooled to r.t. and filtered through Celite® before addition of sat. NH_4Cl and sat. $NaHCO_3$. The mixture was extracted with 4x dichloromethane and the combined organic phases dried over $MgSO_4$, filtered and concentrated under reduced pressure, yielding a mixture of (3*S*, 4*S*, 5*S*, 6*R*/*S*)-3,4,5-tri-*O*-benzyl-6-hydroxy-1,8-nonadiene (**61**) as a transparent oil.

Ac_2O (30 mL, 317 mmol, 17.8 eq.) and cat. DMAP were added to crude (3*S*, 4*S*, 5*S*, 6*R*/*S*)-3,4,5-tri-*O*-benzyl-6-hydroxy-1,8-nonadiene (**61**) in pyridine (30 mL). The mixture was stirred under a nitrogen atmosphere at r.t. for 5 h before additional Ac_2O (10 mL, 106 mmol, 5.93 eq.) and cat. DMAP were added. After additional 2 h the reaction was cooled on a water bath and quenched by slow addition of water (20 mL), then diluting with ethyl acetate. The organic phase was washed with 1 M aq. HCl several times, until

[a] Zinc dust (1.01 g) was mixed with 1M HCl (10 mL) and stirred at r.t for 30 min. The mixture was then filtered and the zinc dust was washed with water and diethyl ether. At last the zinc dust was dried under high vacuum with a heat gun.

the water phase was acidic. The organic phase was further washed with sat. $NaHCO_3$, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography (pentane/ethyl acetate 30:1), yielding (3*S*, 4*S*, 5*S*, 6*R*/*S*)-3,4,5-tri-*O*-benzyl-6-hydroxy-1,8-nonadiene (**62**) as a transparent oil (59%, 2.61 g).

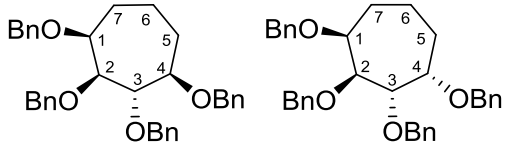
Grubbs-Hoveyda Catalyst 2nd Generation (5x4 mg, 1 mol%) was added portion wise over 24 h to (3*S*, 4*S*, 5*S*, 6*R*/*S*)-3,4,5-tri-*O*-benzyl-6-*O*-acetyl-1,8-nonadiene (**62**) (2.25 g, 4.52 mmol) in dichloromethane (190 mL). The mixture was stirred under inert atmosphere at reflux overnight. The reaction was then quenched by cooling to r.t. and concentrated under reduced pressure. The crude compound was purified by flash column chromatography (pentane/ethyl acetate 20:1) yielding (3*S*,4*S*, 5*S*, 6*R*/*S*)-3,4,5-tri-*O*-benzyl-6-*O*-acetyl-cycloheptene (**63**) as a clear oil (83%, 1.77 g).

A catalytic amount of solid sodium was added to (3*S*, 4*S*, 5*S*, 6*R*/*S*)-3,4,5-tri-*O*-benzyl-6-*O*-acetyl-cycloheptene (**63**) (1.77 g, 3.75 mmol) in anhydrous methanol (40.0 mL) and stirred under inert atmosphere at r.t. overnight. The reaction was then concentrated under reduced pressure and purified by flash column chromatography (pentane/ethyl acetate 20:1 → 10:1), yielding (3*S*, 4*S*, 5*S*, 6*R*)-3,4,5-tri-*O*-benzyl-6-hydroxyl-cycloheptene (**64**) and (3*S*, 4*S*, 5*S*, 6*S*)-3,4,5-tri-*O*-benzyl-6-hydroxyl-cycloheptene (**65**) as a clear oil (Combined yield of R- and S-isomer: 87%, 1.41 g).

(3*S*, 4*S*, 5*S*, 6*R*)-3,4,5-tri-*O*-benzyl-6-hydroxyl-cycloheptene (**64**)**:** R_f (pentane/ethyl acetate 9:1) 0.26; $[\alpha]_D^{295K}$ +38.1 (c 1.0, MeOH); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.32-7.17 (m, 15H, Ar*H*), 5.81 (dd, 1H, $J_{1,2}$ 11.2 Hz, $J_{2,3}$ 4.2 Hz, H2), 5.72 (m, 1H, H1), 4.69 and 4.51 (d, each 1H, $J_{A,B}$ 11.8 Hz, CH_AH_BPh), 4.59 (d, 1H, $J_{A,B}$ 12.1 Hz, C$H_{A/B}$Ph), 4.57 (d, 2H, $J_{A,B}$ 12.1 Hz, C$H_{A/B}$Ph), 4.46 (d, 1H, $J_{2,3}$ 4.2 Hz, H3), 4.45 (d, 1H, $J_{A,B}$ 12.1 Hz, C$H_{A/B}$Ph), 3.87-3.75 (m, 3H, H4, H5, H6), 3.43 (bs, 1H, O*H*), 2.49-2.38 (m, 2H, H7). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C 138.3 (Ar*C*), 138.2 (Ar*C*), 138.1 (Ar*C*), 131.6 (C2), 128.5 (Ar*C*/C1), 128.4 (Ar*C*/C1), 128.3 (Ar*C*/C1), 127.8, 127.7, 127.6, 127.5, 127.4, 127.3 (Ar*C*), 81.1 (C4/5), 80.1 (C4/5), 76.4 (C3), 73.0 (*C*H$_2$Ph), 72.8 (*C*H$_2$Ph), 71.2 (*C*H$_2$Ph), 69.5 (C6), 30.5 (C7). IR: ν_{max} (cm^{-1}): 3507 (w, OH), 3063 (w, sp^2 carbons), 3029 (w, sp^2 carbons), 2867 (w, sp^3 carbons), 1067 (s, ether), 1044 (s), 1027 (s), 732 (s, aromat/double bond), 695 (s, aromat/double bond). HRMS (ESI): calcd. for $C_{28}H_{30}O_4Na$: 453.2042; found 453.2025.

65: R_f (pentane/ethyl acetate 5:1) 0.24; $[\alpha]_D^{295K}$ +55.0 (c 1.0, MeOH); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.35-7.12 (m, 15H, Ar*H*), 5.78-5.69 (m, 2H, H1, H1/2), 4.71 (d, 1H, $J_{A,B}$ 12.2 Hz, C$H_{A/B}$Ph), 4.55 (2x d, 2H, $J_{A,B}$ 12.2 Hz, $J_{A,B}$ 11.5 Hz, 2x C$H_{A/B}$Ph), 4.54 (d, 1H, $J_{A,B}$ 12.2 Hz, CH$_{A/B}$Ph), 4.48 (bs, 1H, H3), 4.42 (d, 1H, $J_{A,B}$ 11.5 Hz CH$_{A/B}$Ph), 4.36 (d, 1H, $J_{A,B}$ 12.2 Hz, C$H_{A/B}$Ph), 3.88 (m, 2H, H4/5, H6), 3.80 (dd, 1H, $J_{3,4/4,5/5,6}$ 5.3 Hz, $J_{3,4/4,5/5,6}$ 2.7 Hz, H4/5), 2.59-2-49 (m, 1H, H7$_{A/B}$), 2.20 (bs, 1H, O*H*), 2.11-2.03 (m, 1H, H7$_{A,B}$). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C 138.6 (Ar*C*), 138.5 (Ar*C*), 138.1 (Ar*C*), 132.6 (C1/2), 128.6, 128.4, 128.3, 127.9, 127.8, 127.7, 127.6, 127.5, 127.4 (Ar*C*), 126.8 (C1/2), 81.6 (C4/5), 78.7 (C4/5), 76.0 (C3), 73.5 (*C*H$_2$Ph), 72.8 (*C*H$_2$Ph), 71.2 (*C*H$_2$Ph), 67.5 (C6), 31.1 (C7). IR: ν_{max} (cm^{-1}): 3449 (w, OH), 3029 (w, sp^2 carbons), 2865 (w, sp^3 carbons), 1069 (s, ether), 1026 (s), 733 (s, aromat/double bond), 694 (s, aromat/double bond). HRMS (ESI): calcd. for $C_{28}H_{30}O_4Na$: 453.2042; found 453.2026.

(1*S*, 2*S*, 3*S*, 4*R*)-1,2,3,5-Tetra-*O*-benzyl-cycloheptane (68) and (1*S*, 2*S*, 3*S*, 4*S*)-1,2,3,4-Tetra-*O*-benzyl-cycloheptane (69):



68 **69**

Step 1:

(3*S*, 4*S*, 5*S*, 6*R*)-3,4,5-Tri-*O*-benzyl-6-hydroxy-cycloheptene (**64**) (53.7 mg, 0.12 mmol) was dissolved in anhydrous DMF (0.45 mL), then added 60% NaH in mineral oil (15.0 mg, 0.38 mmol, 3.2 eq.) at 0 °C and stirred for 10 min. before addition of BnBr (0.06 mL, 0.51 mmol, 4.3 eq.). The mixture was allowed to reach r.t. and quenched after 2 h by cooling to 0 °C and slow addition of ice water. The mixture was extracted with 4x ethyl acetate and the combined organic phases washed with water and brine, dried over $MgSO_4$, filtered and concentrated under reduced pressure. The crude product was purified by flash column chromatography (pentane/ethyl acetate 15:1), yielding (3*S*, 4*S*, 5*S*, 6*R*)-3,4,5,6-tetra-*O*-benzyl-cycloheptene (**66**) as a clear oil (75%, 47.0 mg).

(3*S*, 4*S*, 5*S*, 6*S*)-3,4,5,6-tetra-*O*-benzyl-cycloheptene (**67**) was prepared by the same procedure (Step 1) starting from (3*S*, 4*S*, 5*S*, 6*S*)-3,4,5-tri-*O*-benzyl-6-hydroxy-cycloheptene (**65**) (48 mg, 0.11 mmol), yielding compound **67** as a clear oil (71%, 41.0 mg).

Step 2:

Triethylamine (0.01 mL, 0.07 mmol, 0.77 eq.) and a catalytic amount of $Pd(OH)_2/C$ was added to (3*S*, 4*S*, 5*S*, 6*R*)-3,4,5,6-tetra-*O*-benzyl-cycloheptene (**66**) (47.0 mg, 0.09 mmol) in anhydrous methanol (0.50 mL) and stirred under inert atmosphere at r.t.. The atmosphere was exchanged with H_2 (g) (1 atm.) and the reaction stirred rapidly for 40 min. The reaction was quenched by exchanging the atmosphere to nitrogen, filtration through Celite® and concentration under reduced pressure. The crude product was purified by flash column chromatography (pentane/ethyl acetate 30:1), yielding (1*S*, 2*S*, 3*S*, 4*R*)-1,2,3,4-tetra-*O*-benzyl-cycloheptane (**68**) as a clear oil (40%, 18.9 mg)

(1*S*, 2*S*, 3*S*, 4*S*)-1,2,3,4-Tetra-*O*-benzyl-cycloheptane (**69**) was prepared by the same procedure (Step 2) starting from (3*S*, 4*S*, 5*S*, 6*S*)-3,4,5,6-tetra-*O*-benzyl-cycloheptene (**67**) (41 mg, 0.08 mmol) yielding **69** as a clear oil (79%, 32.5 mg).

(1*S*, 2*S*, 3*S*, 4*R*)-1,2,3,4-tetra-*O*-benzyl-cycloheptane (**68**)**:** R_f (pentane/ethyl acetate 30:1) 0.26; $[\alpha]_D^{295K}$ -4.68 (c 1.0, MeOH); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.37-7.17 (m, 20H, Ar*H*), 4.74-4.42 (6x d, 8H, 8x C$H_{A/B}$Ph), 3.90 (d, 1H, $J_{1,2/2,3}$ 2.9 Hz, H2), 3.71 (dd, 1H, $J_{3,4}$ 6.6 Hz, $J_{2,3}$ 2.9 Hz, H3), 3.54 (dd, 1H, $J_{1,7A}$ 11.3 Hz, $J_{1,2/1,7B}$ 2.9 Hz, H1), 3.35 (ddd, 1H, $J_{4,5A}$ 10.6 Hz, $J_{3,4}$ 6.6 Hz, $J_{4,5B}$ 1.6 Hz, H4), 2.15-1.69 (m, 5H, H5, H6$_{A/B}$, H7), 1.35-1.21 (m, 1H, H6$_{A/B}$). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C 138.9 (Ar*C*), 138.8 (Ar*C*), 138.7 (Ar*C*), 138.5 (Ar*C*), 128.4, 128.3, 128.2, 127.9, 127.7, 127.6, 127.4, 127.3, 127.2 (Ar*C*), 85.9 (C4), 83.8 (C3), 79.8 (C2), 79.3 (C1), 72.7 (*C*H_2Ph), 72.4 (*C*H_2Ph), 71.5 (*C*H_2Ph), 70.9 (*C*H_2Ph), 28.2 (C5/7), 28.1 (C5/7), 23.7 (C6). IR: ν_{max} (cm^{-1}): 3029 (w, sp^2 carbons), 2860 (w, sp^3 carbons), 1086 (s), 1064 (ether), 732 (s, aromat), 694 (s, aromat). HRMS (ESI): calcd. for $C_{35}H_{38}O_4Na$: 545.2668; found 545.2662.

(1*S*, 2*S*, 3*S*, 4*S*)-1,2,3,4-Tetra-*O*-benzyl-cycloheptane (**69**)**:** R_f (pentane/ethyl acetate 30:1) 0.26; $[\alpha]_D^{295K}$ +3.80 (c 1.0, MeOH); ^{1}H-NMR ($CDCl_3$, 400 MHz): δ_H 7.31-7.16 (m, 20H, Ar*H*), 4.70 and 4.53 (d, each 1H, $J_{A,B}$ 12.1 Hz, CH_AH_BPh), 4.50 and 4.43 (d, each 1H, $J_{A,B}$ 12.2 Hz), 3.83 (bs, 2H, H2, H3), 3.81 (dd, 2H, $J_{1,7/4,5}$ 10.1 Hz, $J_{1,2/1,7/3,4/4,5}$ 2.6 Hz), 1.98-1.85 (m, 2H, H7$_{A/B}$, H5$_{A/B}$), 1.75-1.65 (m, 2H, H7$_{A/B}$, H5$_{A/B}$), 1.65-

1.59 (m, 2H, H6). ^{13}C-NMR ($CDCl_3$, 100 MHz): δ_C 139.1, 128.3, 128.2, 127.6, 127.5, 127.4, 127.3 (Ar*C*), 79.7 (C2, C3), 78.3 (C1, C4), 73.2 (2x *C*H_2Ph), 71.0 (2x *C*H_2Ph), 26.3 (C5, C7), 20.2 (C6). IR: ν_{max} (cm^{-1}): 3062 (w, sp^2 carbons), 3029 (w, sp^2 carbons), 2865 (w, sp^3 carbons), 1086 (s), 1063 (s, ether), 1026 (s), 731 (s, aromat), 694 (s, aromat). HRMS (ESI): calcd. for $C_{35}H_{38}O_4Na$: 545.2668; found 545.2668.

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(2) Chen, C.-T.; Weng, S.-S.; Kao, J.-Q.; Lin, C.-C.; Jan, M.-D. *Org. Lett.* **2005**, *7*, 3343.

(3) Sadeghi-Khomami, A.; Forcada, T. J.; Wilson, C.; Sanders, D. A. R.; Thomas, N. R. *Org. Biomol. Chem.* **2010**, *8*, 1596.

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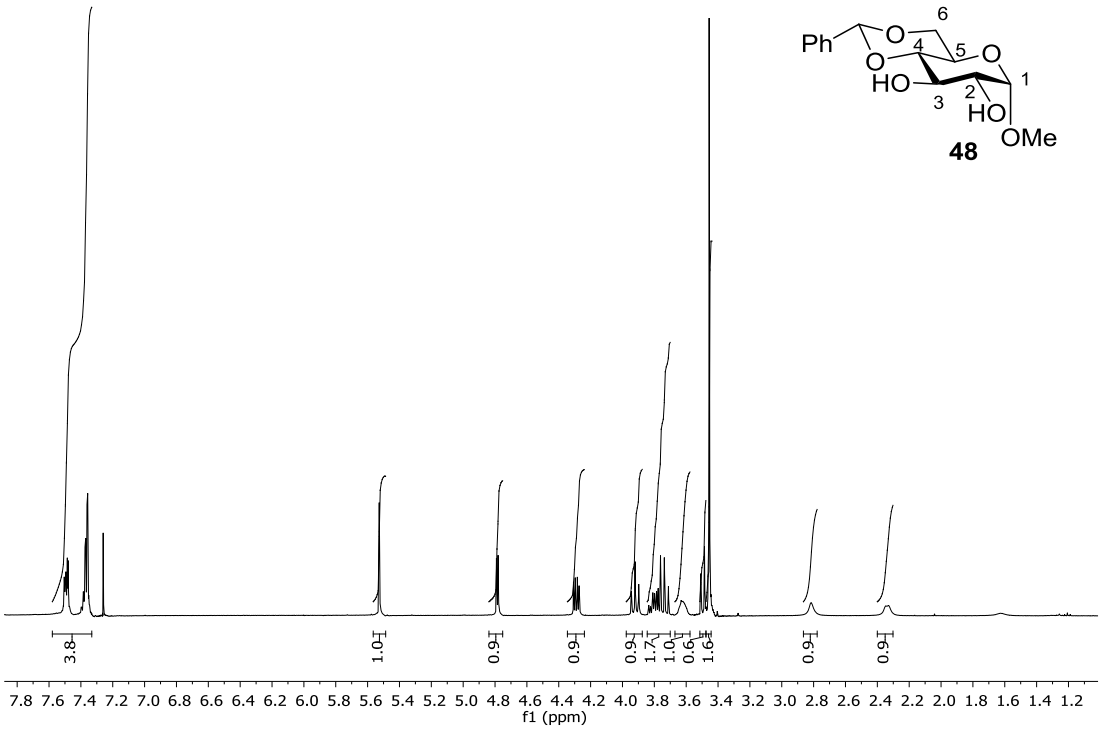
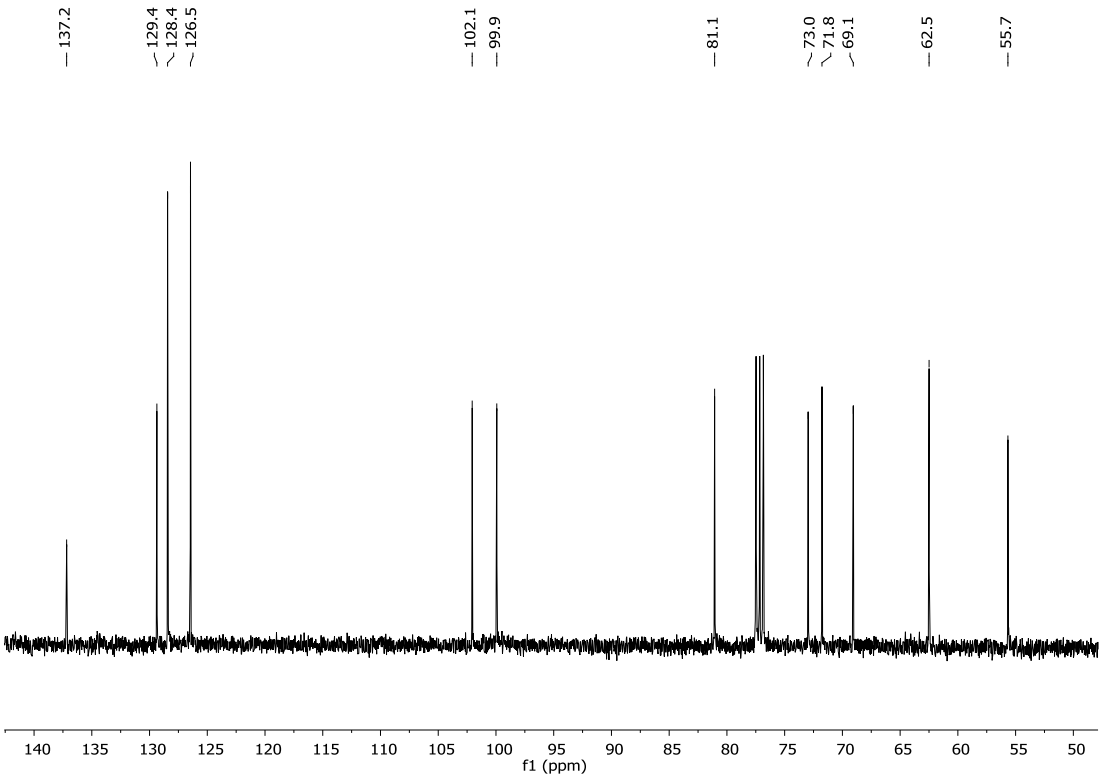
^{1}H-NMR and ^{13}C-NMR spectra for compounds 1-69:

Methyl 4,6-*O*-Benzylidene-α-D-glucopyranoside (48):

^{1}H-NMR (400 MHz, $CDCl_3$):

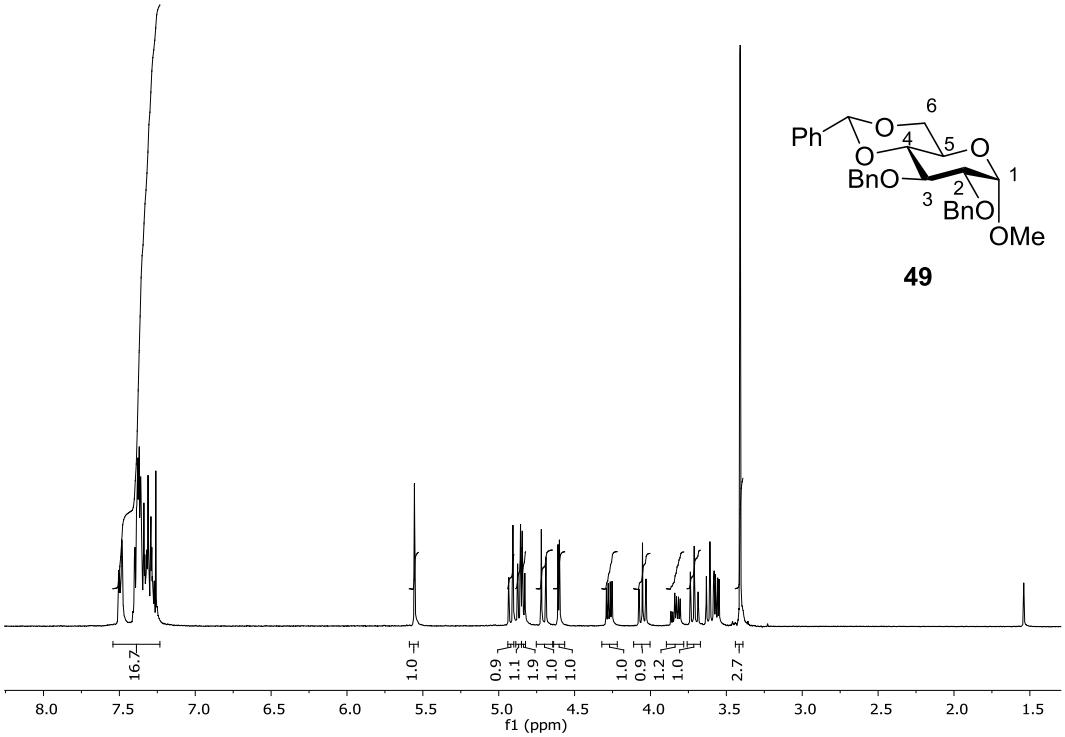


^{13}C-NMR (100 MHz, $CDCl_3$):

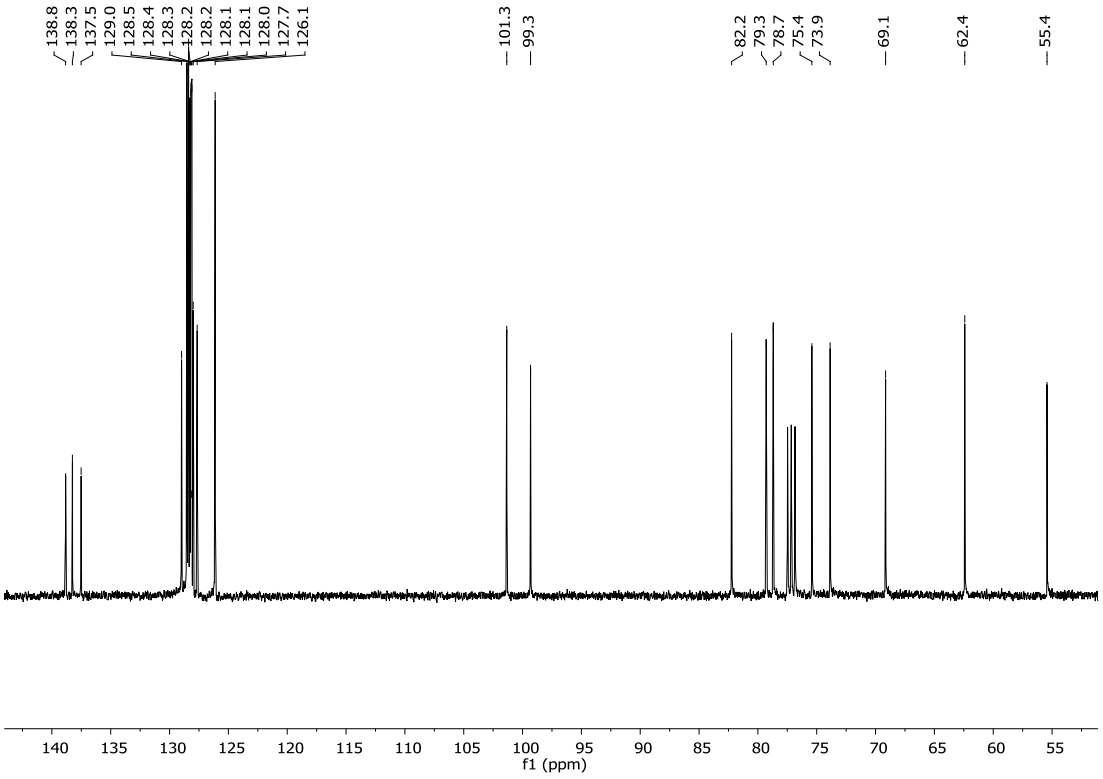


Methyl 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-α-D-glucopyranoside (49):

^{1}H-NMR (400 MHz, $CDCl_3$):

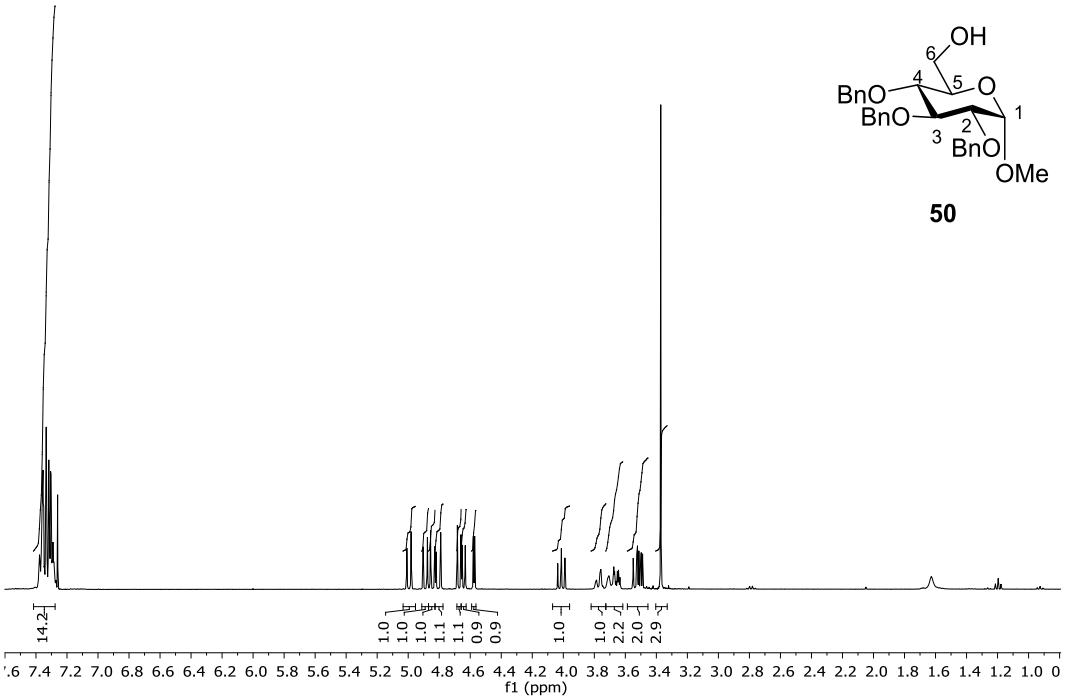


^{13}C-NMR (100 MHz, $CDCl_3$):

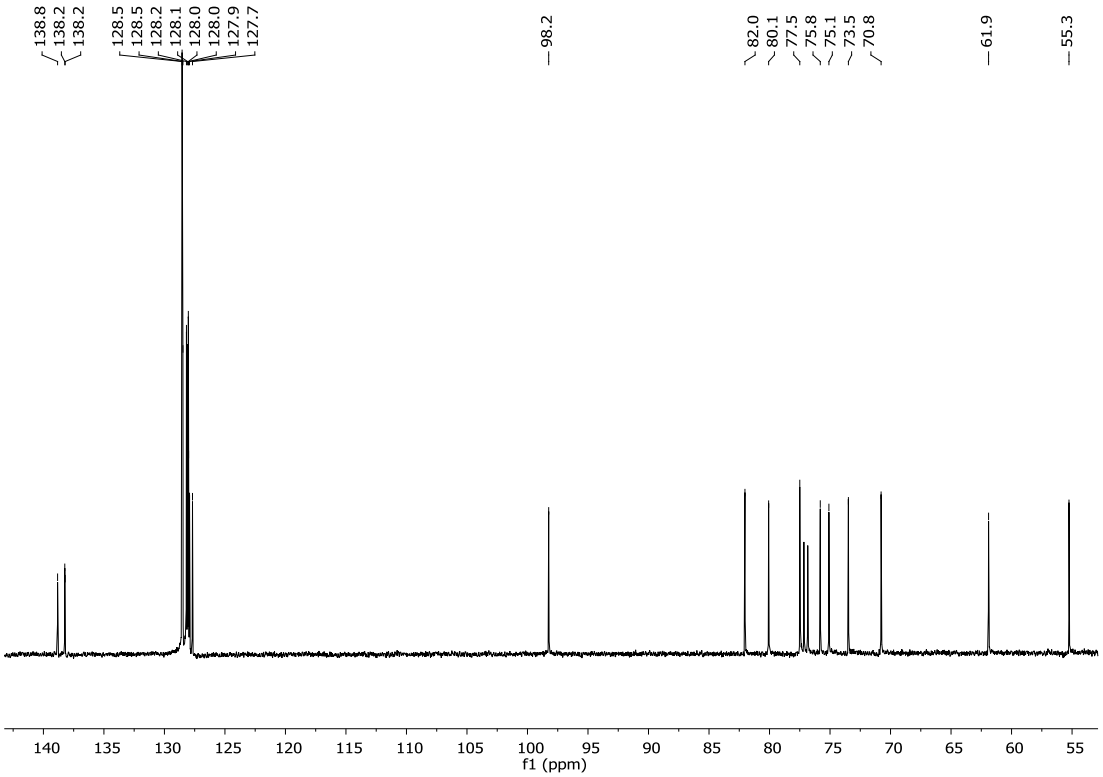


Methyl 2,3,4-tri-*O*-benzyl-α-D-glucopyranoside (50):

^{1}H-NMR (400 MHz, $CDCl_3$):

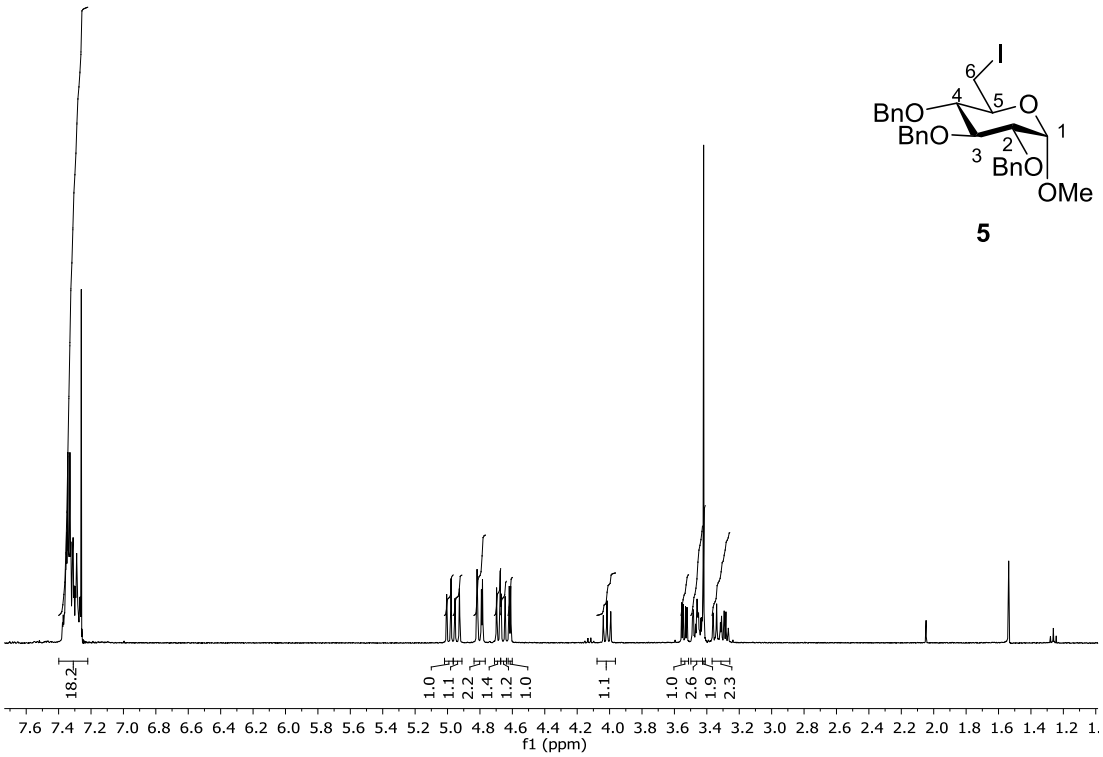


^{13}C-NMR (100 MHz, $CDCl_3$):

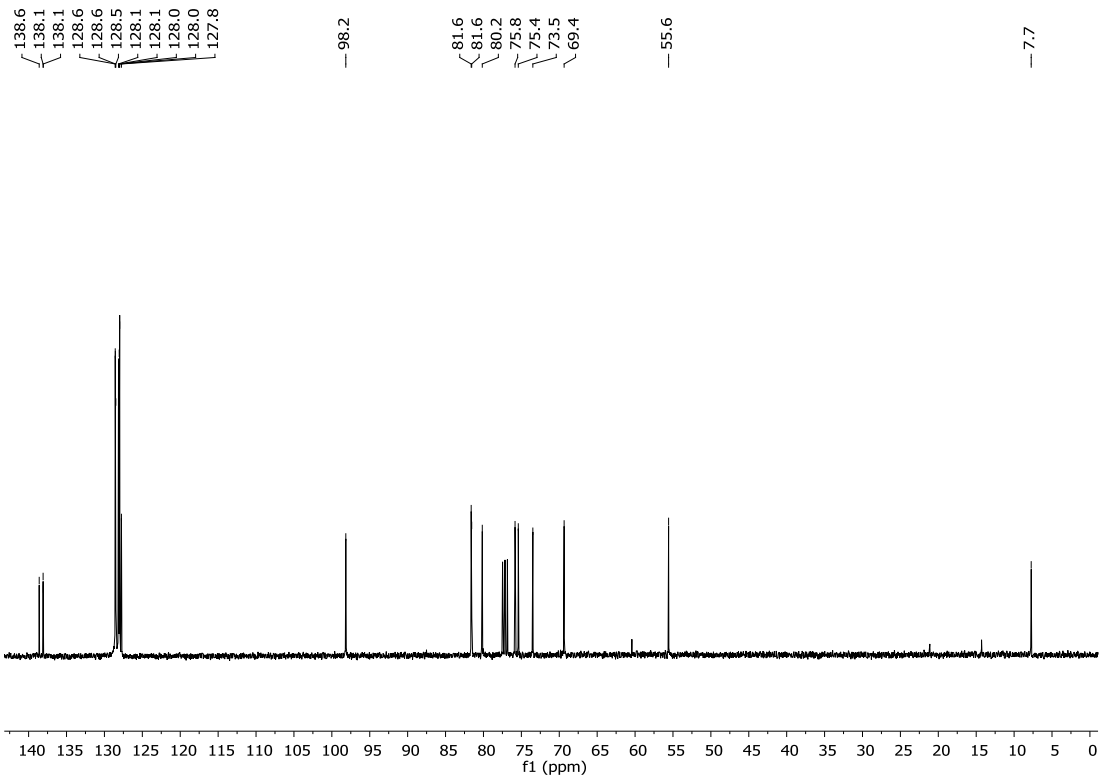


Methyl 2,3,4-tri-*O*-benzyl-6-deoxy-6-iodo-α-D-glucopyranoside (5):

^{1}H-NMR (400 MHz, $CDCl_3$):

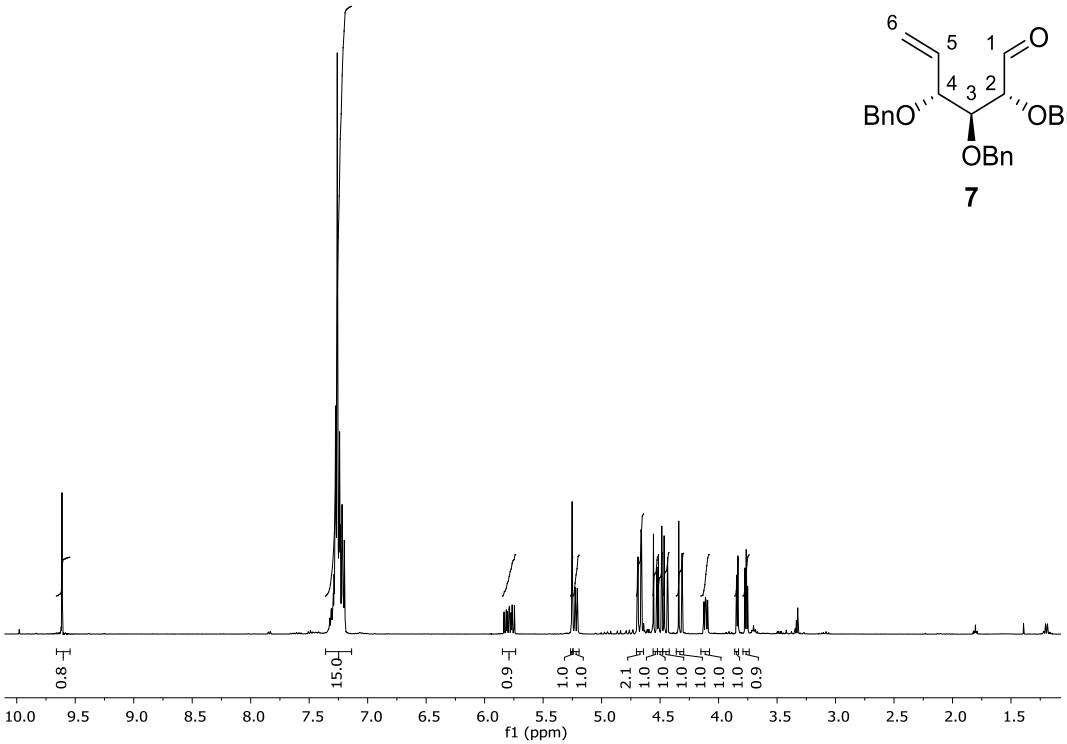


^{13}C-NMR (100 MHz, $CDCl_3$):

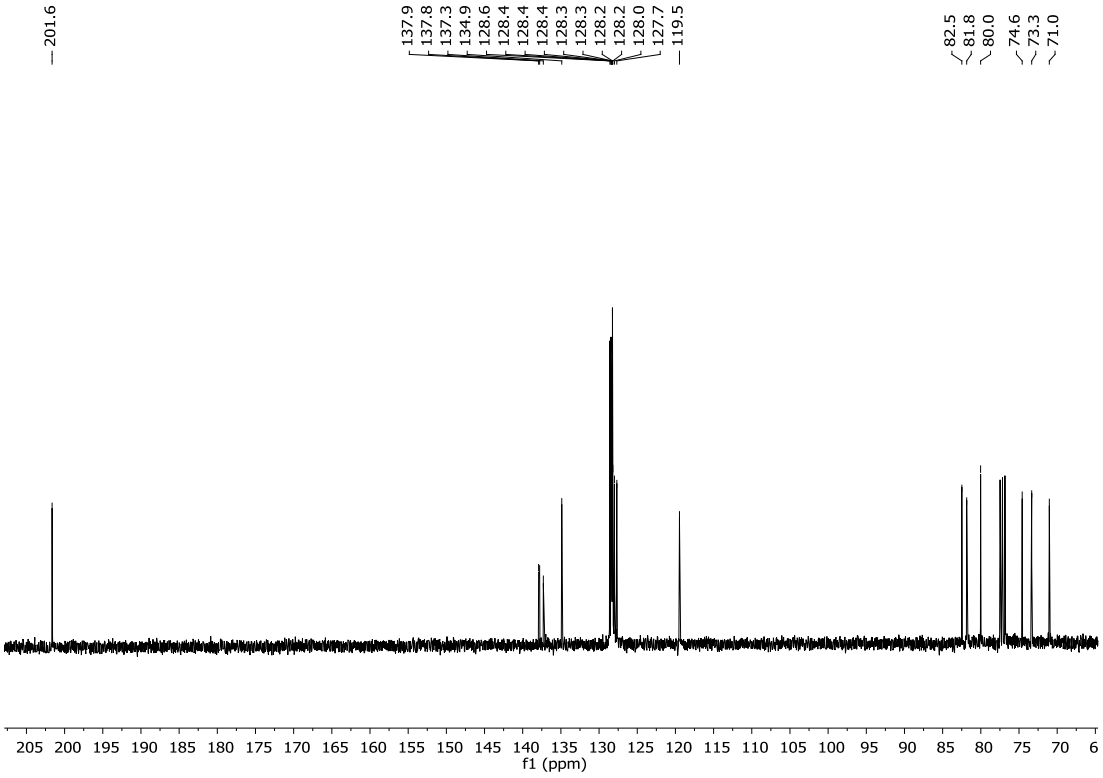


(2*R*,3*S*,4*R*)-2,3,4-Tri-*O*-benzylhex-5-enal (7):

^{1}H-NMR (400 MHz, $CDCl_3$):

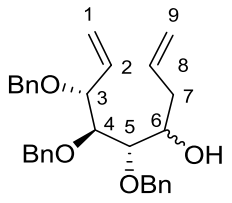


^{13}C-NMR (100 MHz, $CDCl_3$):



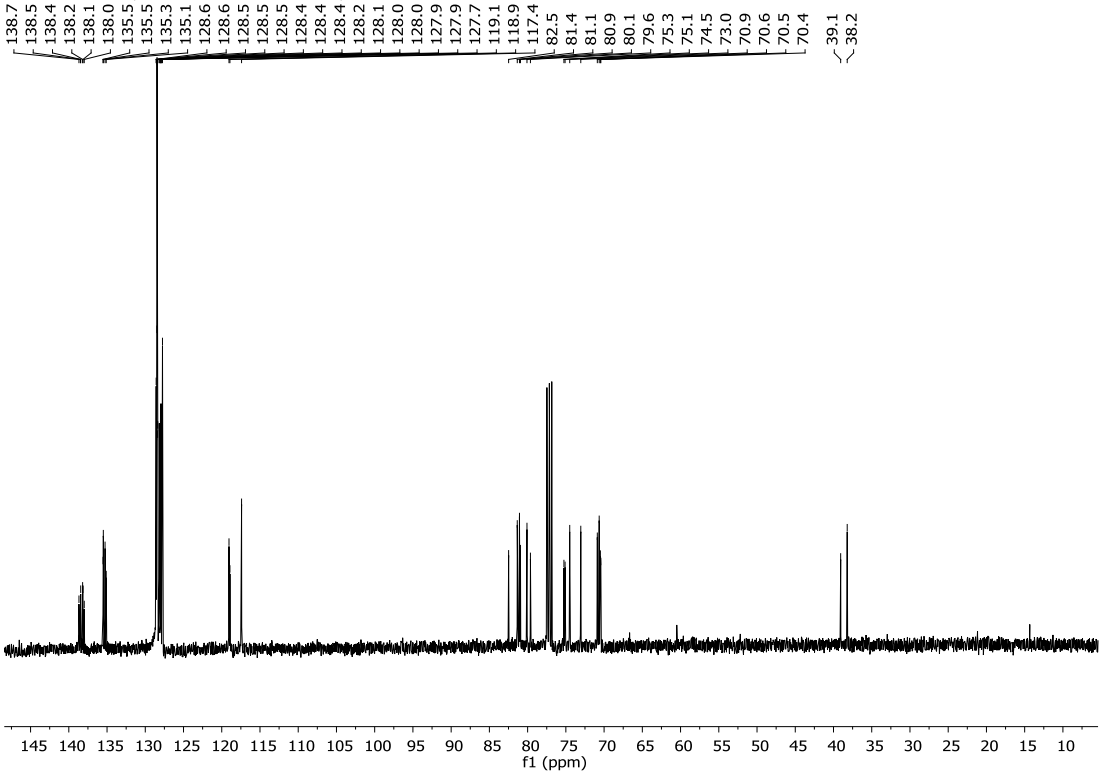
(3*R*, 4*S*, 5*S*, 6*R*/*S*)-3,4,5-Tri-*O*-benzyl-6-hydroxy-1,8-nonadiene (8):

^{1}H-NMR (400 MHz, $CDCl_3$):



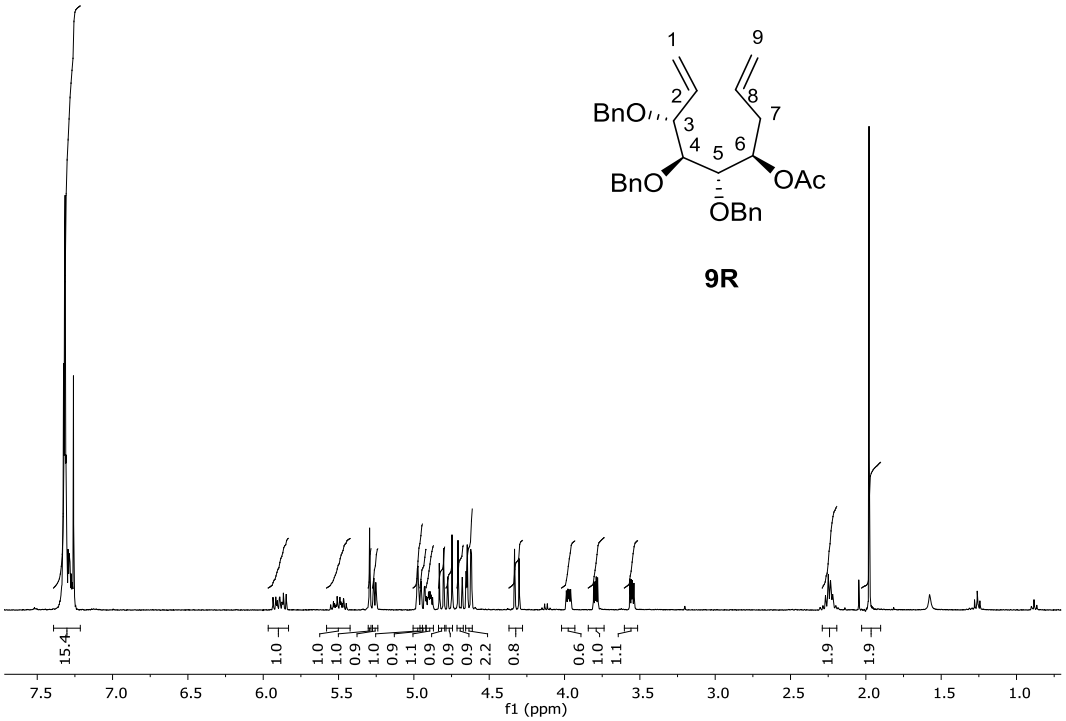
8

^{13}C-NMR (100 MHz, $CDCl_3$):

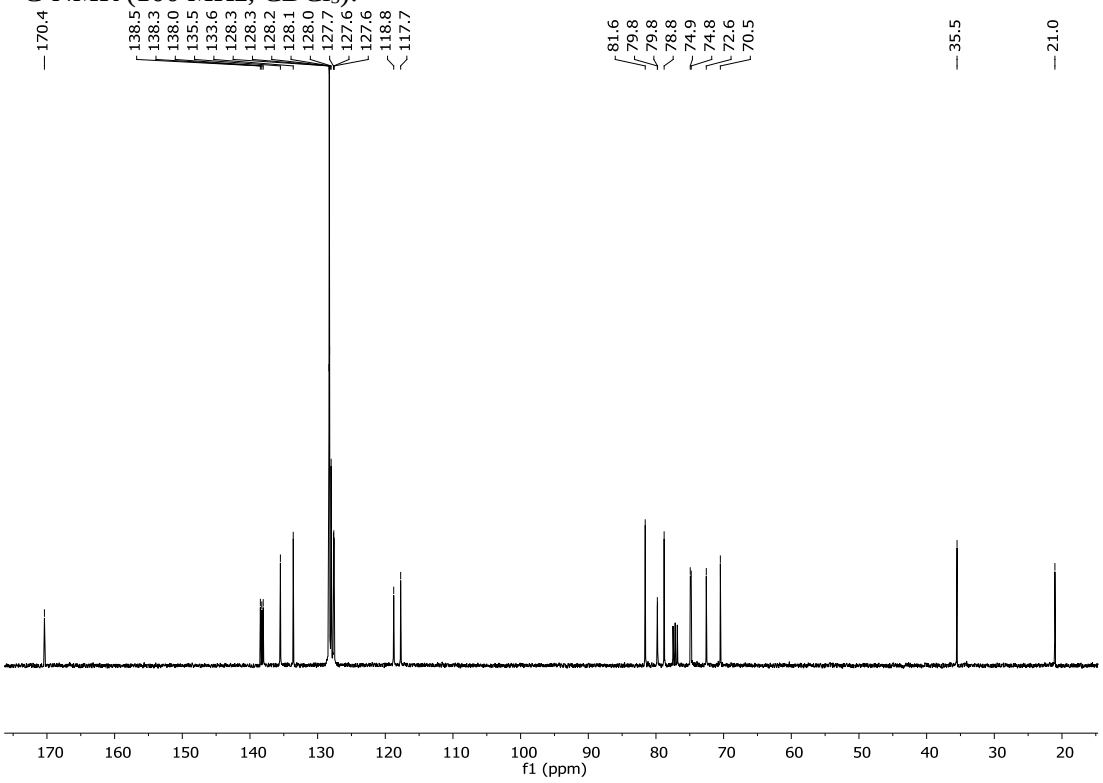


(3*R*, 4*S*, 5*S*, 6*R*)-6-*O*-Acetyl-3,4,5-tri-*O*-benzyl-1,8-nonadiene(9R)

^{1}H-NMR (400 MHz, $CDCl_3$):

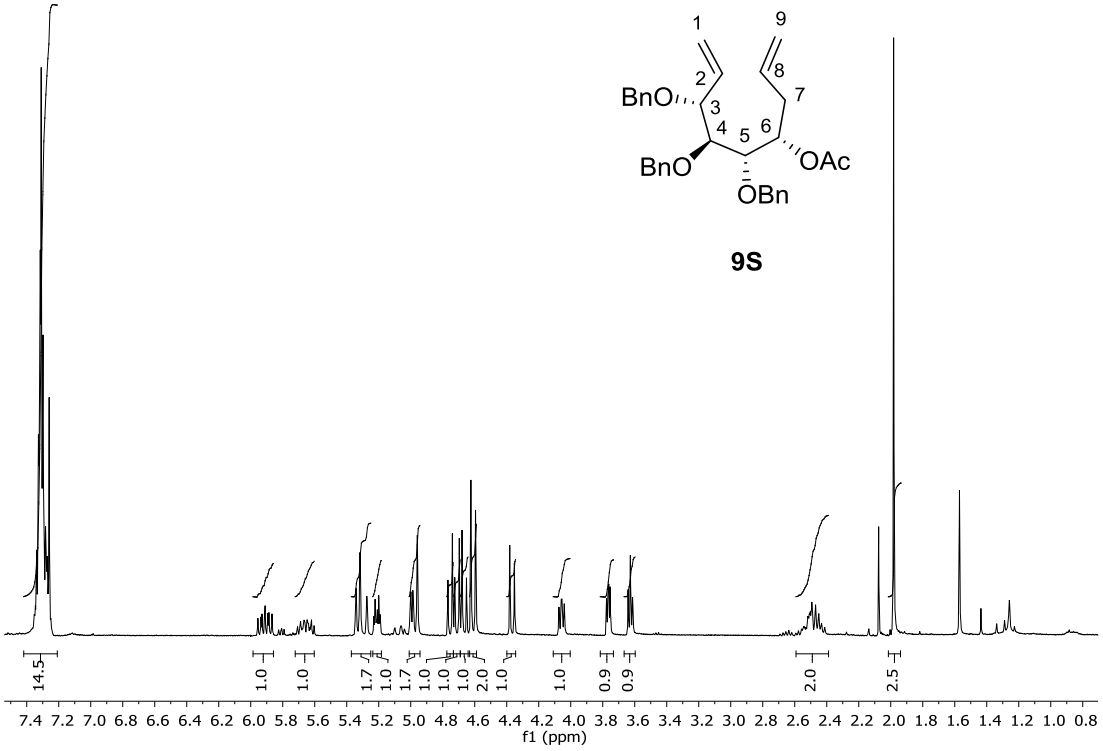


^{13}C-NMR (100 MHz, $CDCl_3$):

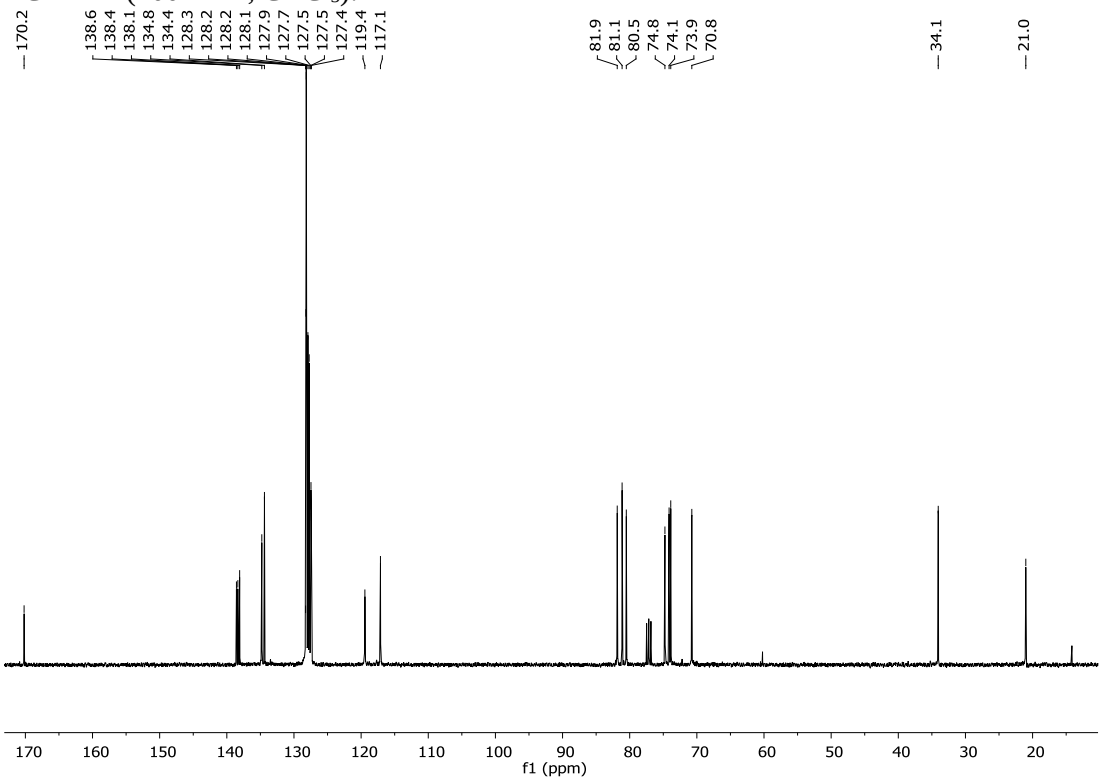


(3*R*, 4*S*, 5*S*, 6*S*)-3,4,5-tri-*O*-benzyl-6-*O*-acetyl-1,8-nonadiene (9S):

^{1}H-NMR (400 MHz, $CDCl_3$):

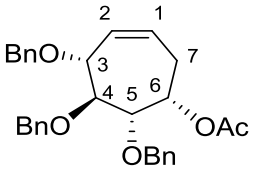


^{13}C-NMR (100 MHz, $CDCl_3$):



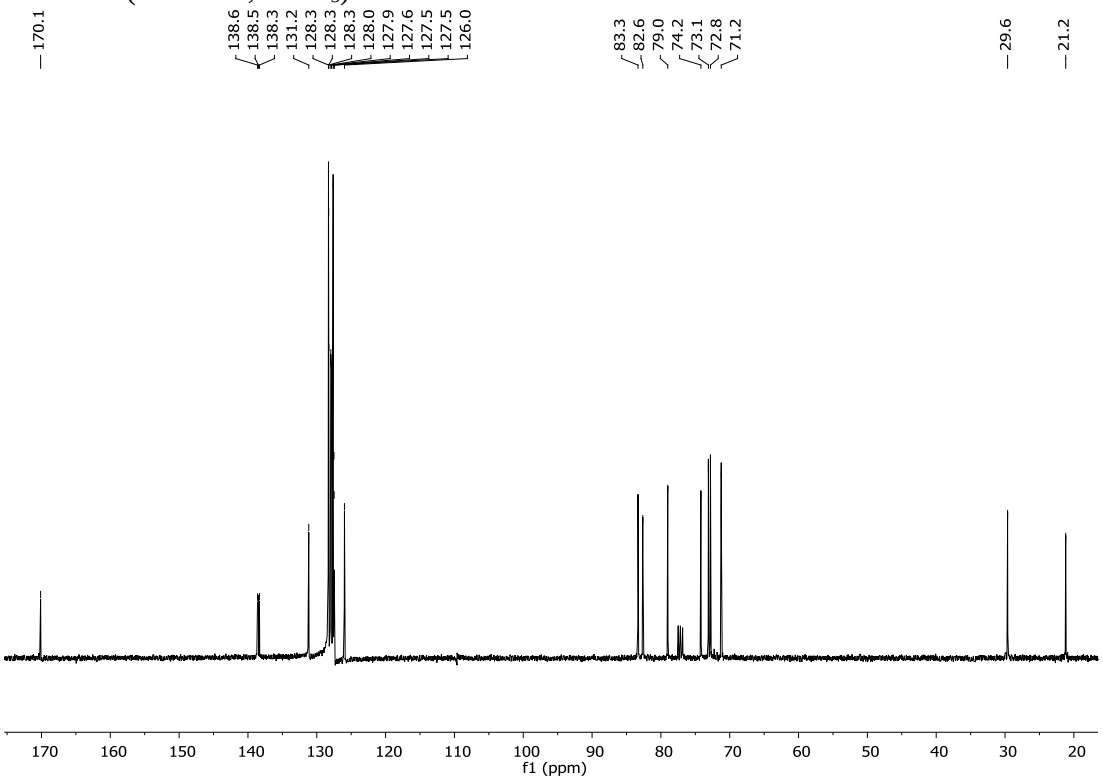
(3*R*, 4*S*, 5*S*, 6*S*)-6-*O*-Acetyl-3,4,5-tri-*O*-benzyl-cycloheptene (10):

^{1}H-NMR (400 MHz, $CDCl_3$):



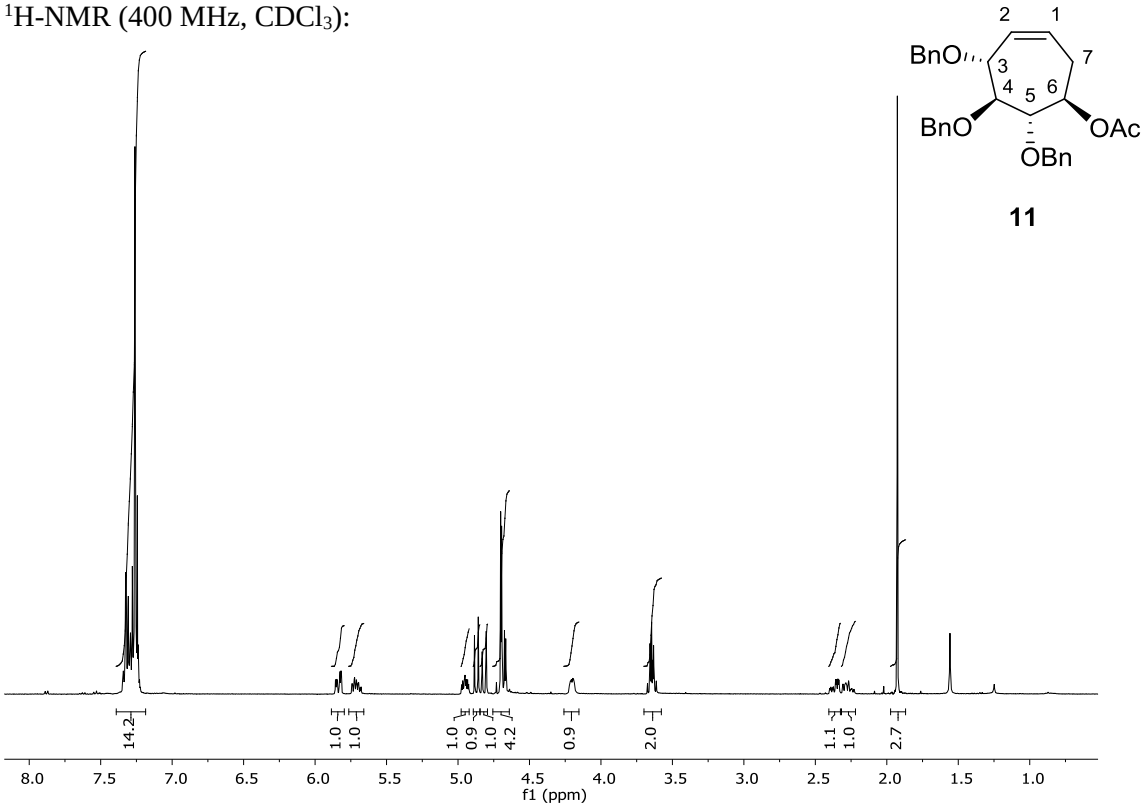
10

^{13}C-NMR (100 MHz, $CDCl_3$):

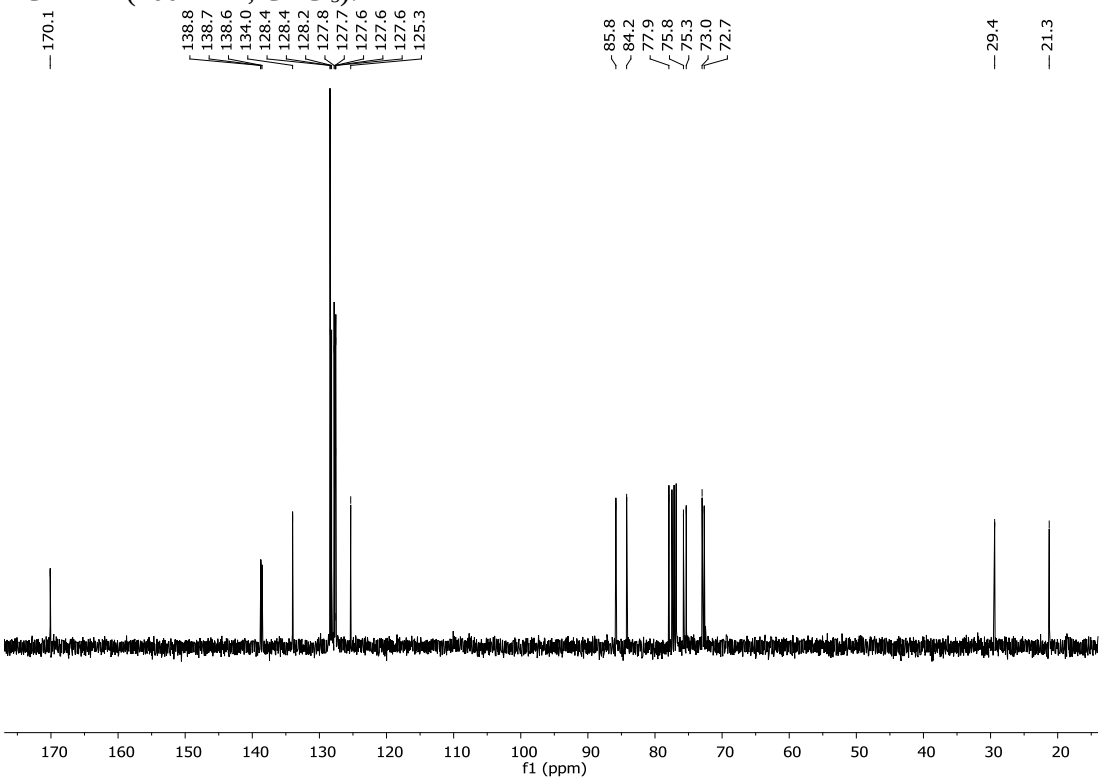


(3*R*, 4*S*, 5*S*, 6*R*)-6-*O*-Acetyl-3,4,5-tri-*O*-benzyl-cycloheptene (11):

^{1}H-NMR (400 MHz, $CDCl_3$):

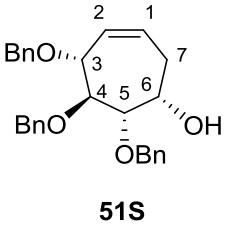


^{13}C-NMR (100 MHz, $CDCl_3$):



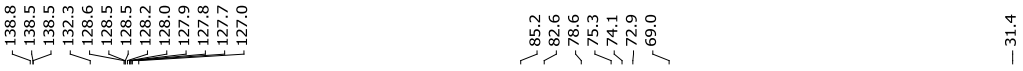
(3*R*, 4*S*, 5*S*, 6*S*)-3,4,5-Tri-*O*-benzyl-6-hydroxy-cycloheptene (51S):

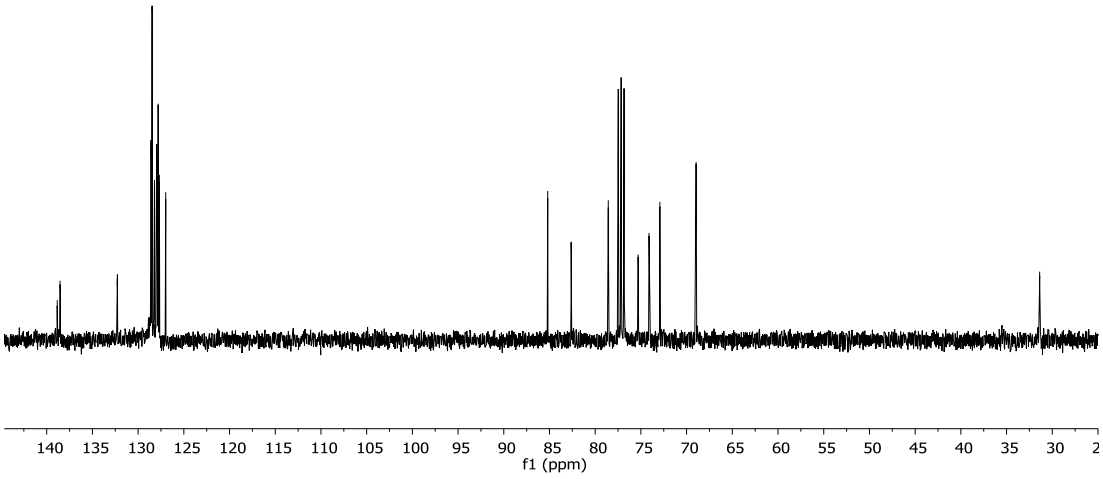
^{1}H-NMR (400 MHz, $CDCl_3$):



51S

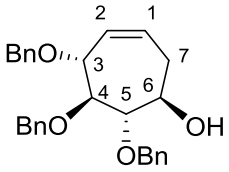
^{13}C-NMR (100 MHz, $CDCl_3$):





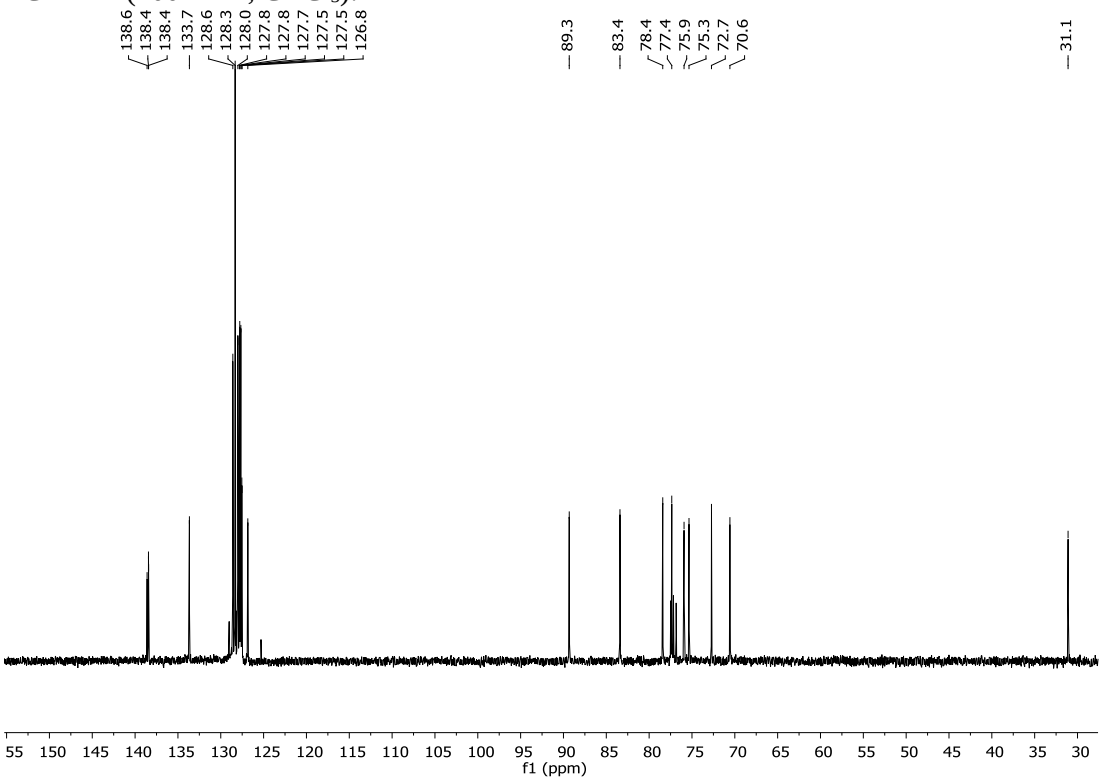
(3*R*, 4*S*, 5*S*, 6*R*)-3,4,5-Tri-*O*-benzyl-6-hydroxy-cycloheptene (51R):

^{1}H-NMR (400 MHz, $CDCl_3$):



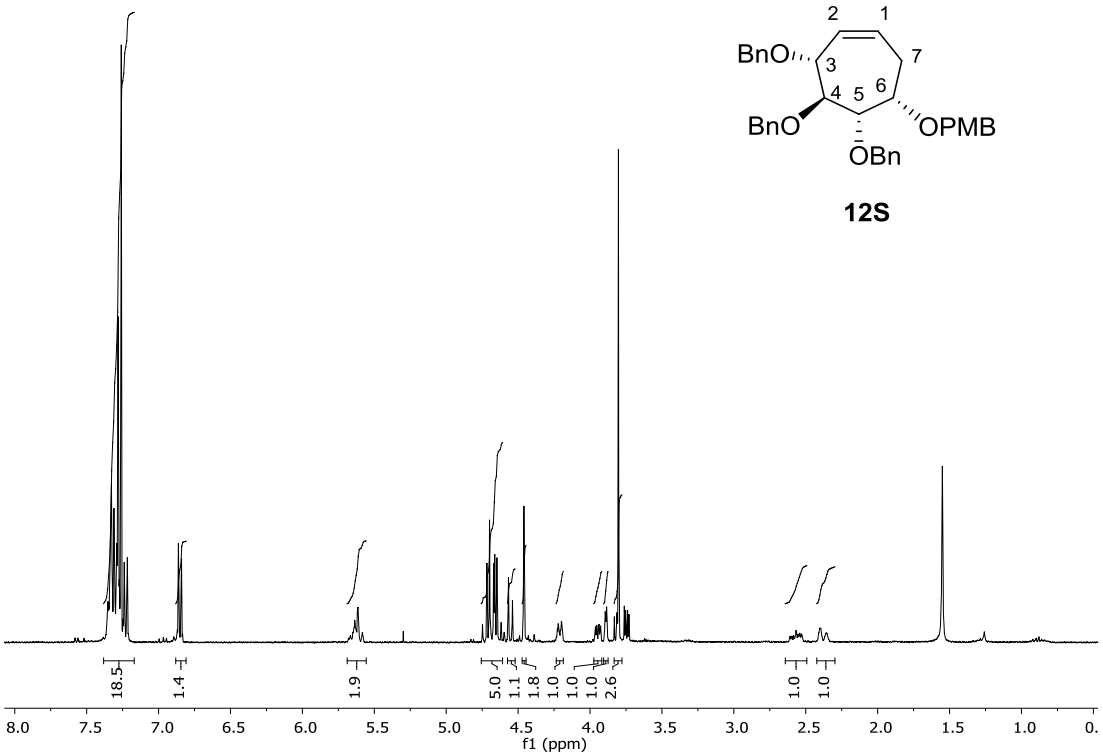
51R

^{13}C-NMR (100 MHz, $CDCl_3$):

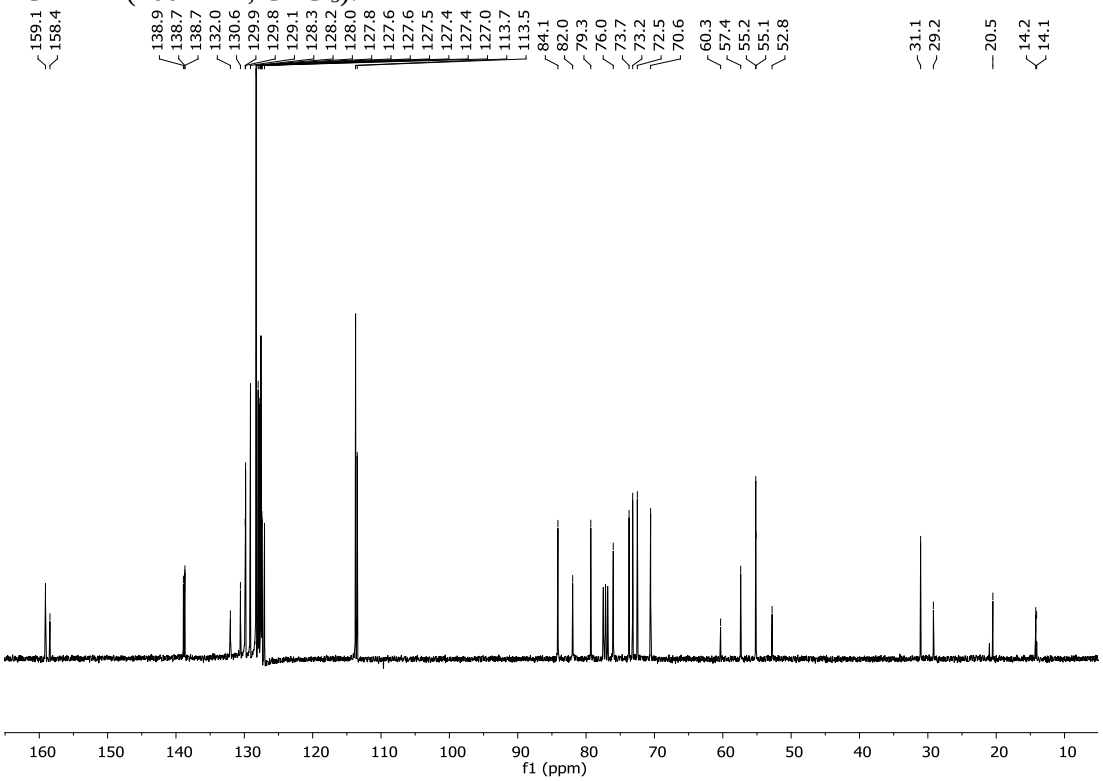


(3*R*, 4*S*, 5*S*, 6*S*)-3,4,5-Tri-*O*-benzyl-6-*O*-*p*-methoxybenzyl-cycloheptene (12S):

^{1}H-NMR (400 MHz, $CDCl_3$):

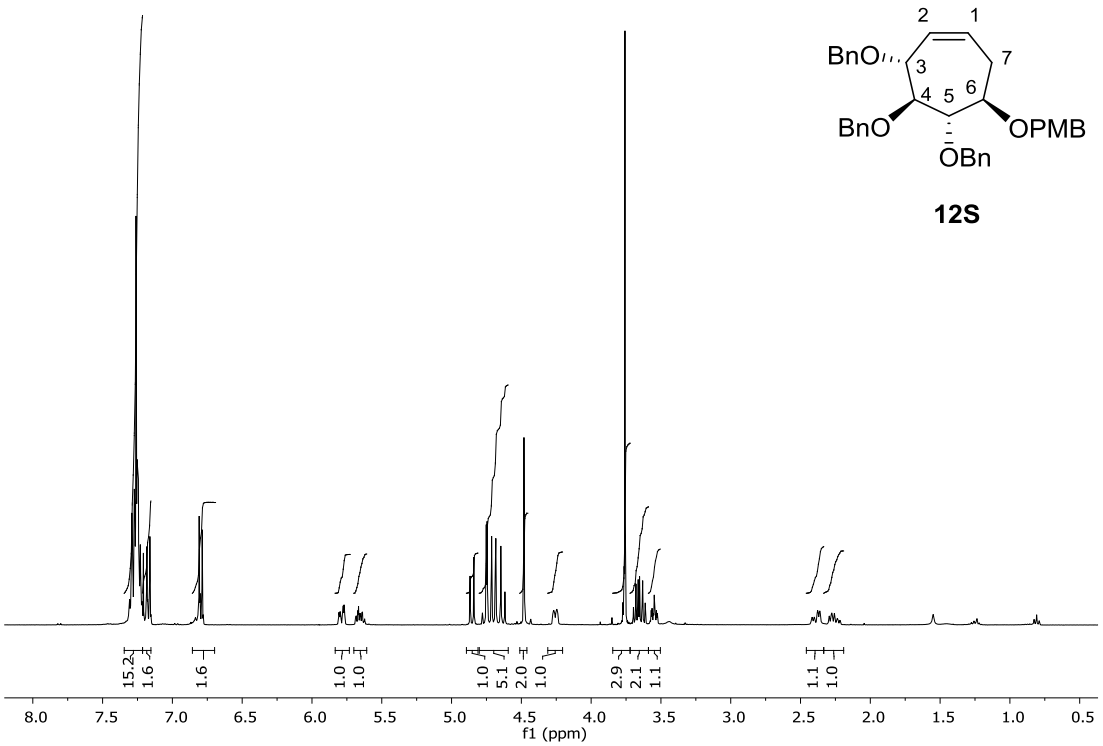


^{13}C-NMR (100 MHz, $CDCl_3$):

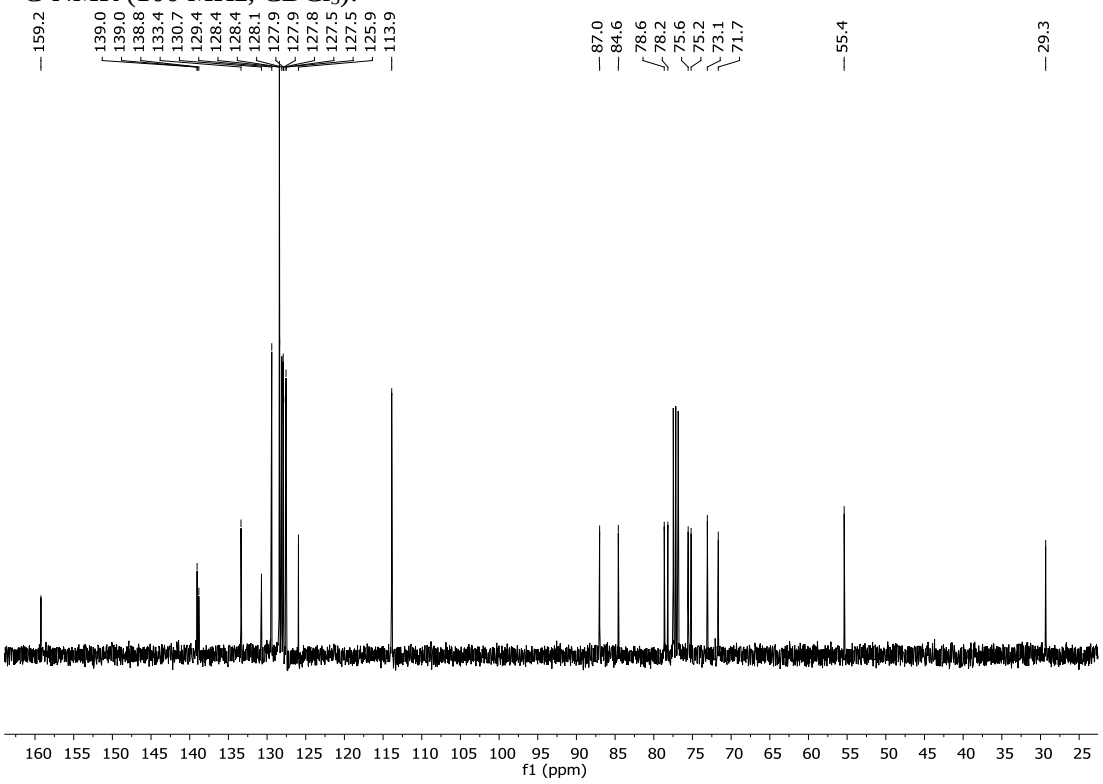


(3*R*, 4*S*, 5*S*, 6*R*)-3,4,5-Tri-*O*-benzyl-6-*O*-*p*-methoxybenzyl-cycloheptene (12R):

^{1}H-NMR (400 MHz, $CDCl_3$):

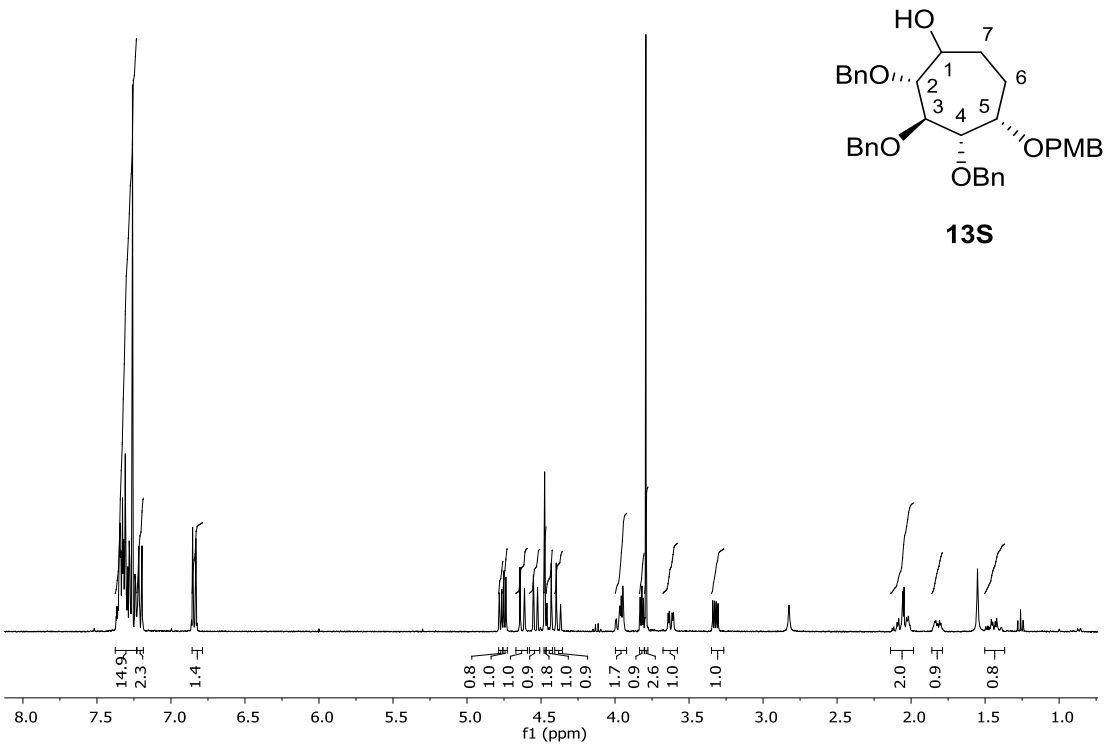


^{13}C-NMR (100 MHz, $CDCl_3$):

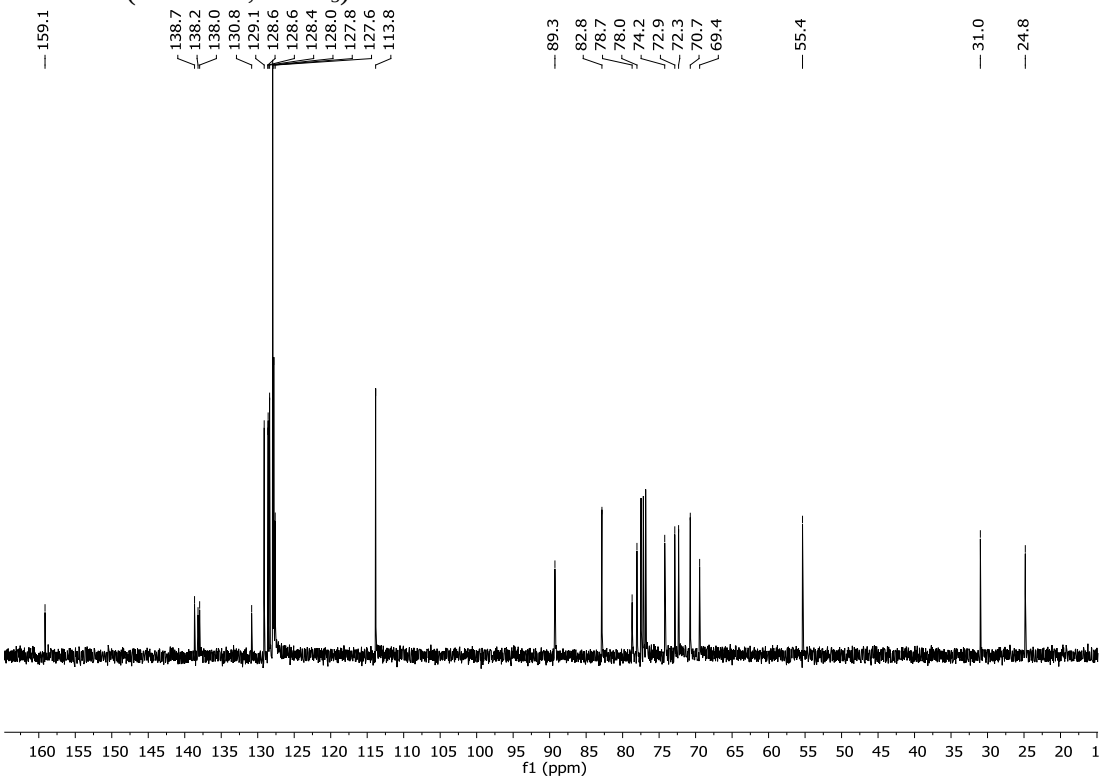


(2*R*, 3*S*, 4*S*, 5*S*)-2,3,4-tri-*O*-benzyl-5-*O*-*p*-methoxybenzyl-cycloheptane-1-ol (13S):

^{1}H-NMR (400 MHz, $CDCl_3$):

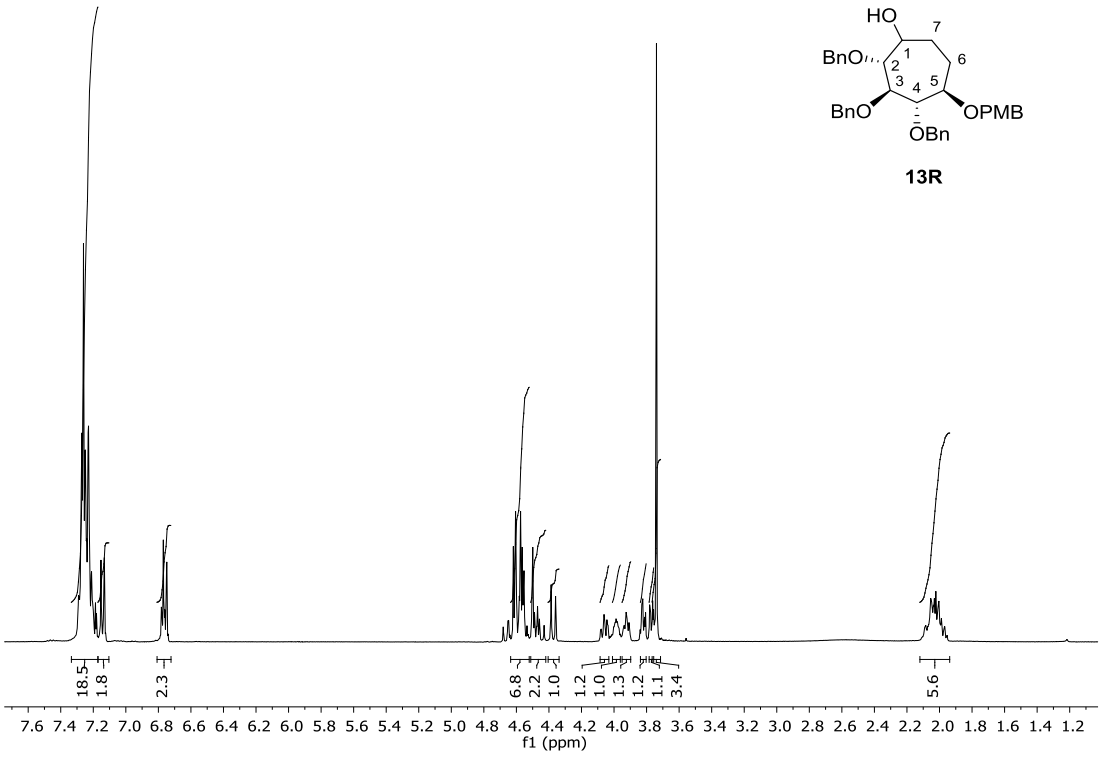


^{13}C-NMR (100 MHz, $CDCl_3$):

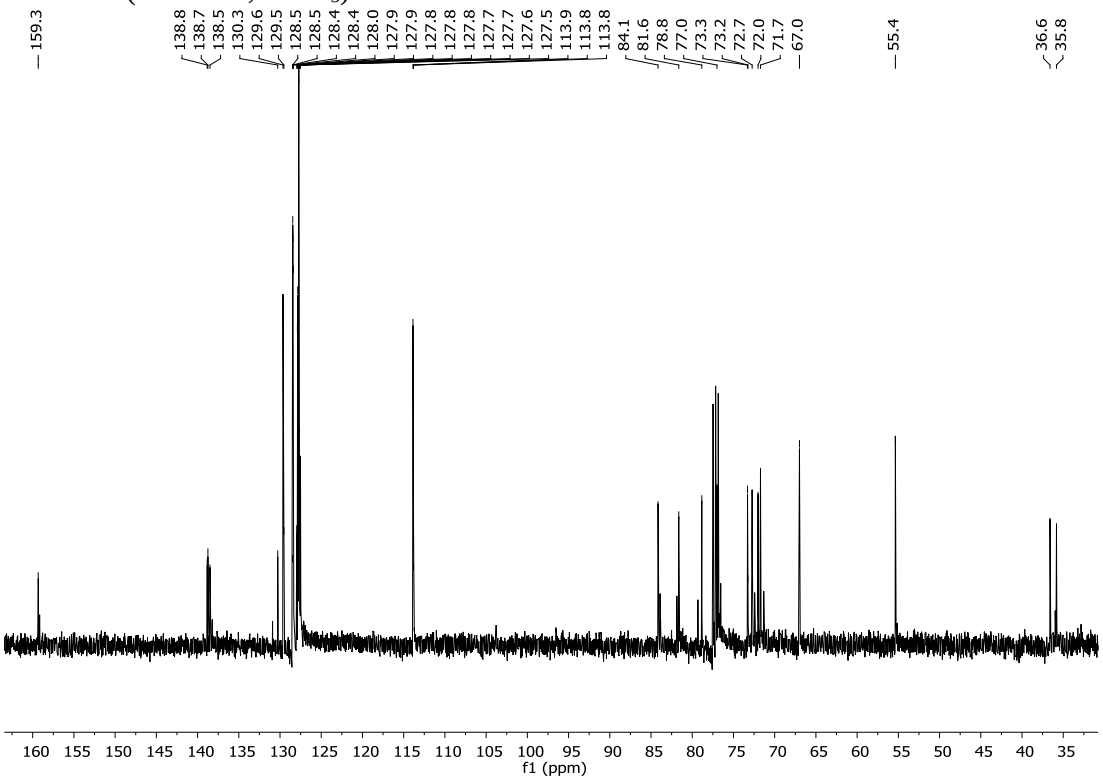


(2*R*, 3*S*, 4*S*, 5*R*)-2,3,4-tri-*O*-benzyl-5-*O*-*p*-methoxybenzyl-cycloheptane-1-ol (13R):

^{1}H-NMR (400 MHz, $CDCl_3$):

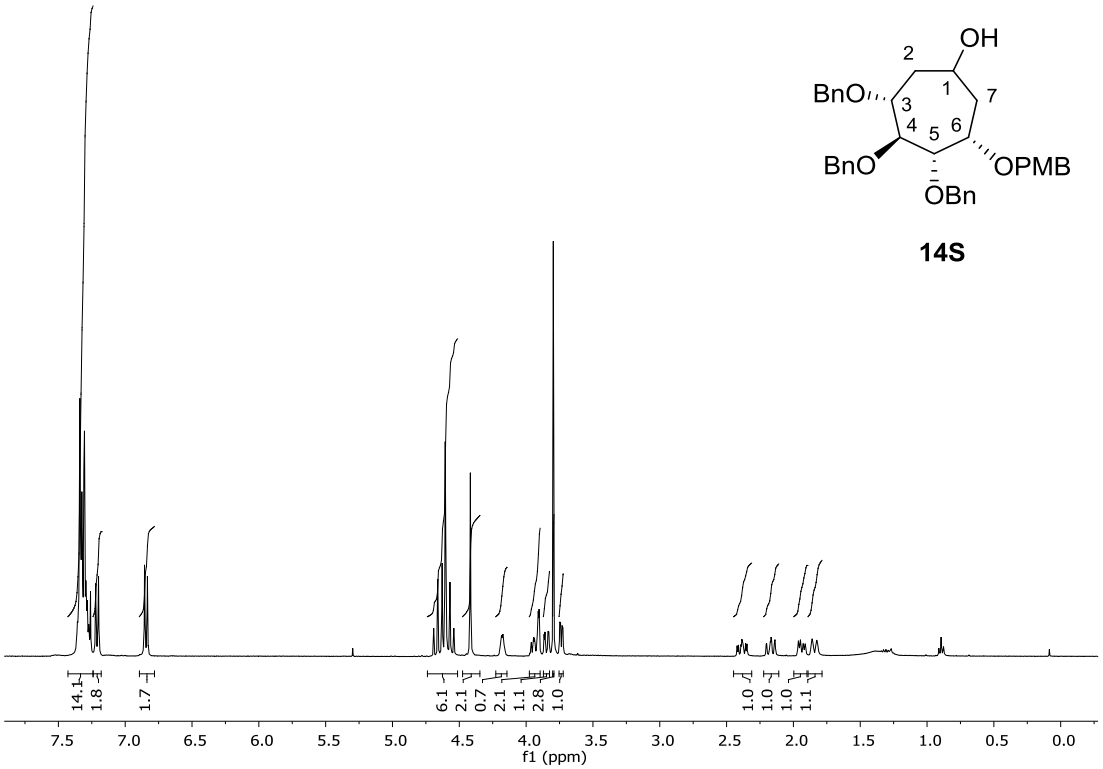


^{13}C-NMR (100 MHz, $CDCl_3$):

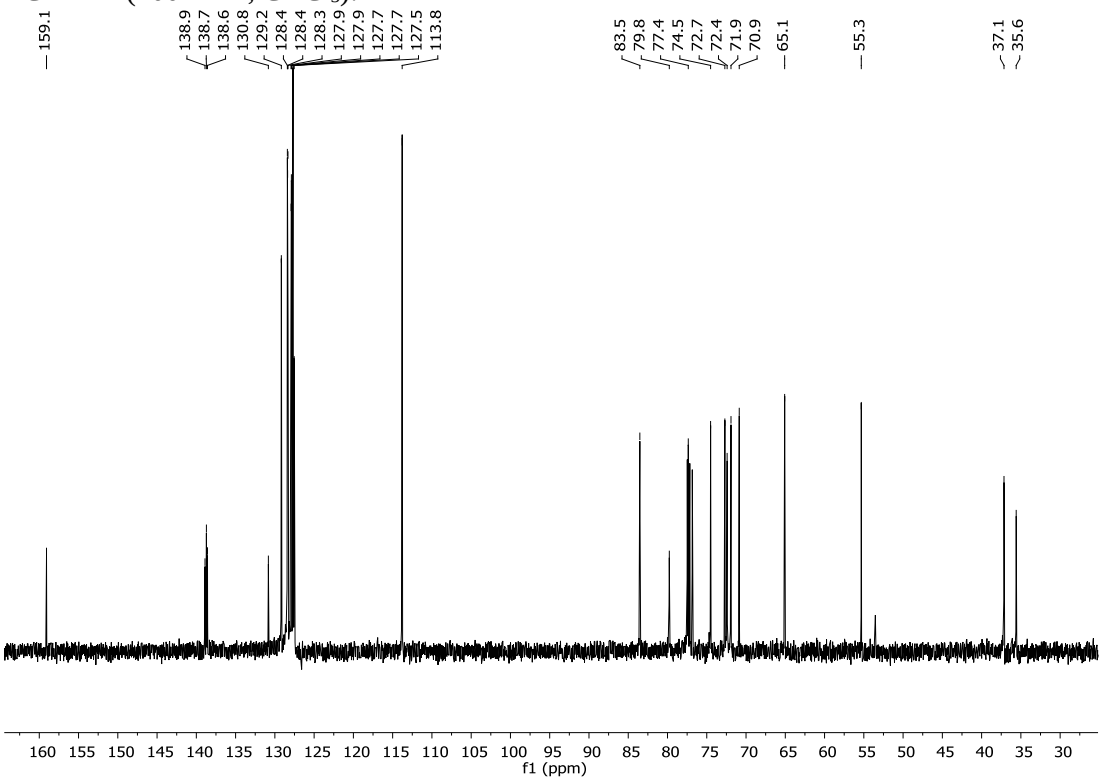


(3*R*, 4*S*, 5*S*, 6*S*)-3,4,5-tri-*O*-benzyl-6-*O*-*p*-methoxybenzyl-cycloheptane-1-ol (14S):

^{1}H-NMR (400 MHz, $CDCl_3$):

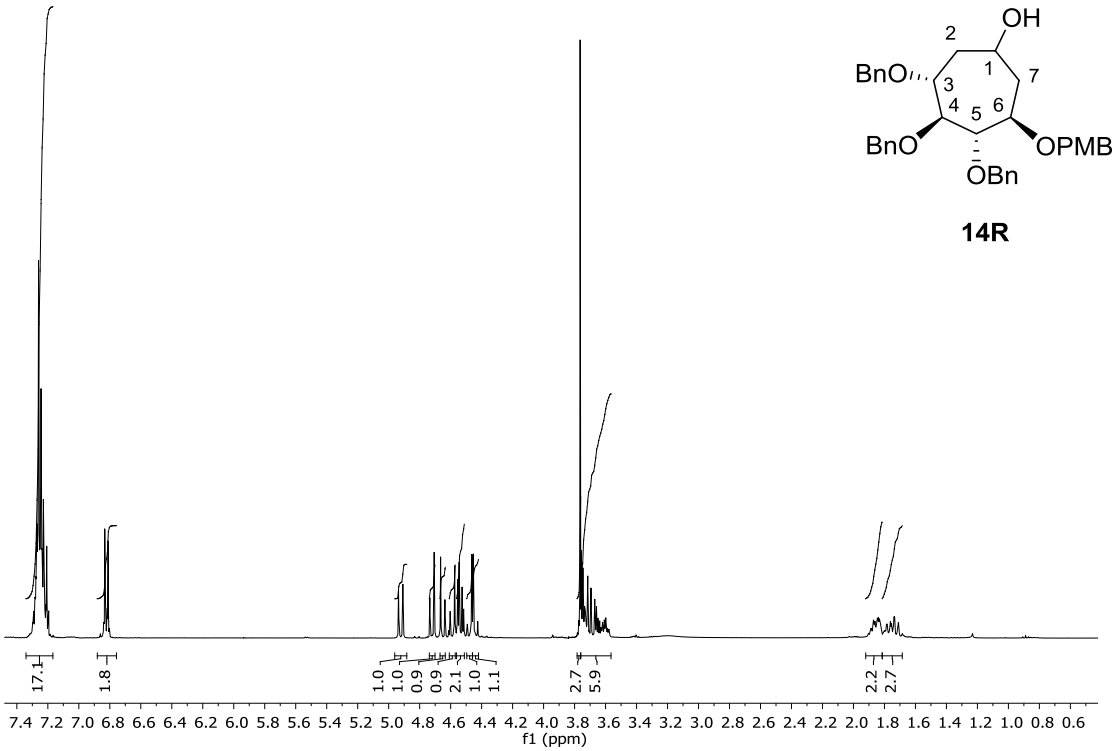


^{13}C-NMR (100 MHz, $CDCl_3$):

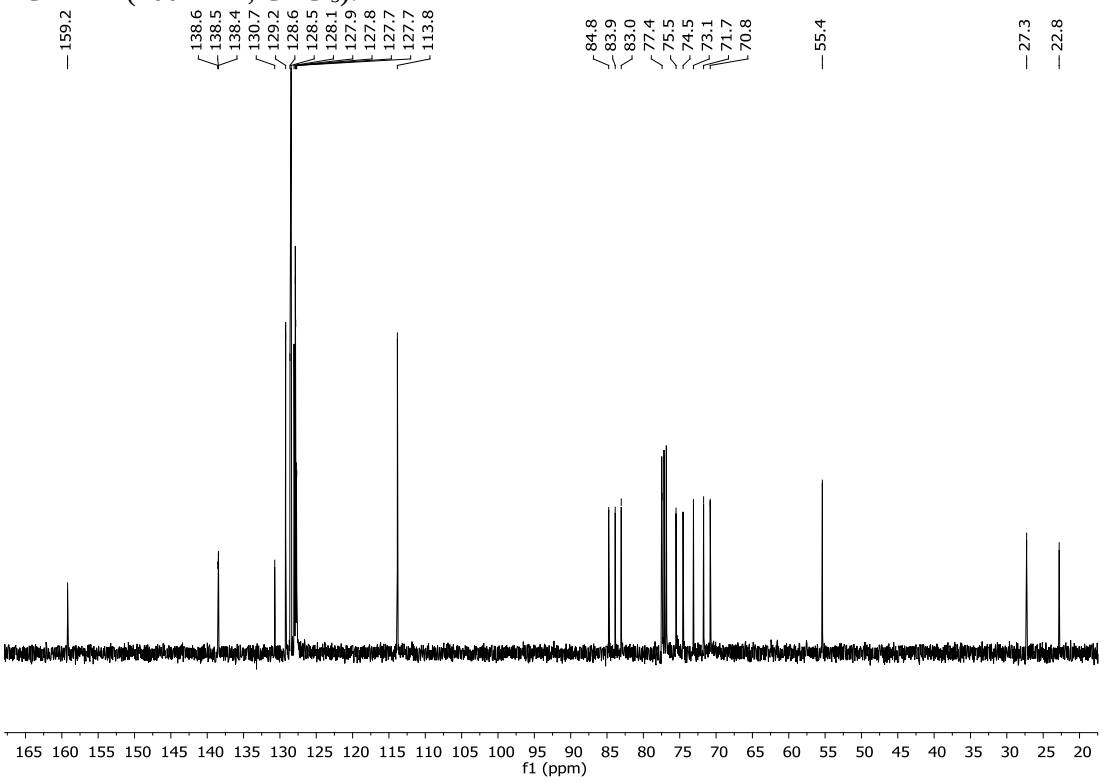


(3*R*, 4*S*, 5*S*, 6*R*)-3,4,5-tri-*O*-benzyl-6-*O*-*p*-methoxybenzyl-cycloheptane-1-ol (14R):

^{1}H-NMR (400 MHz, $CDCl_3$):

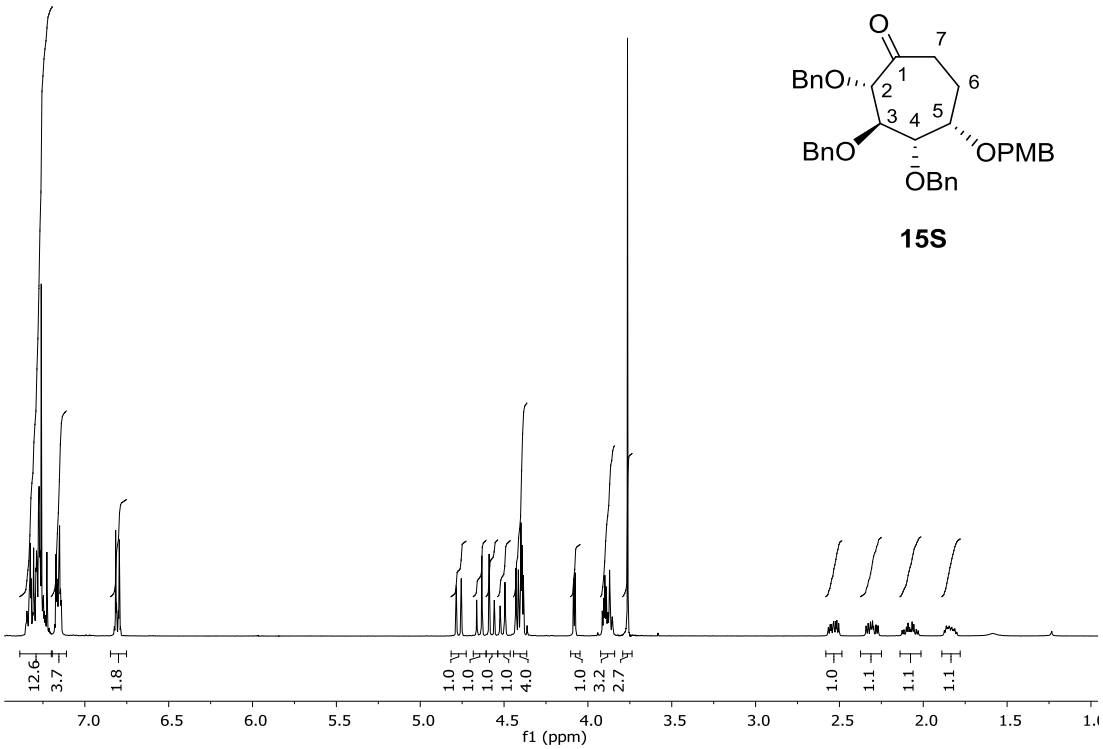


^{13}C-NMR (100 MHz, $CDCl_3$):

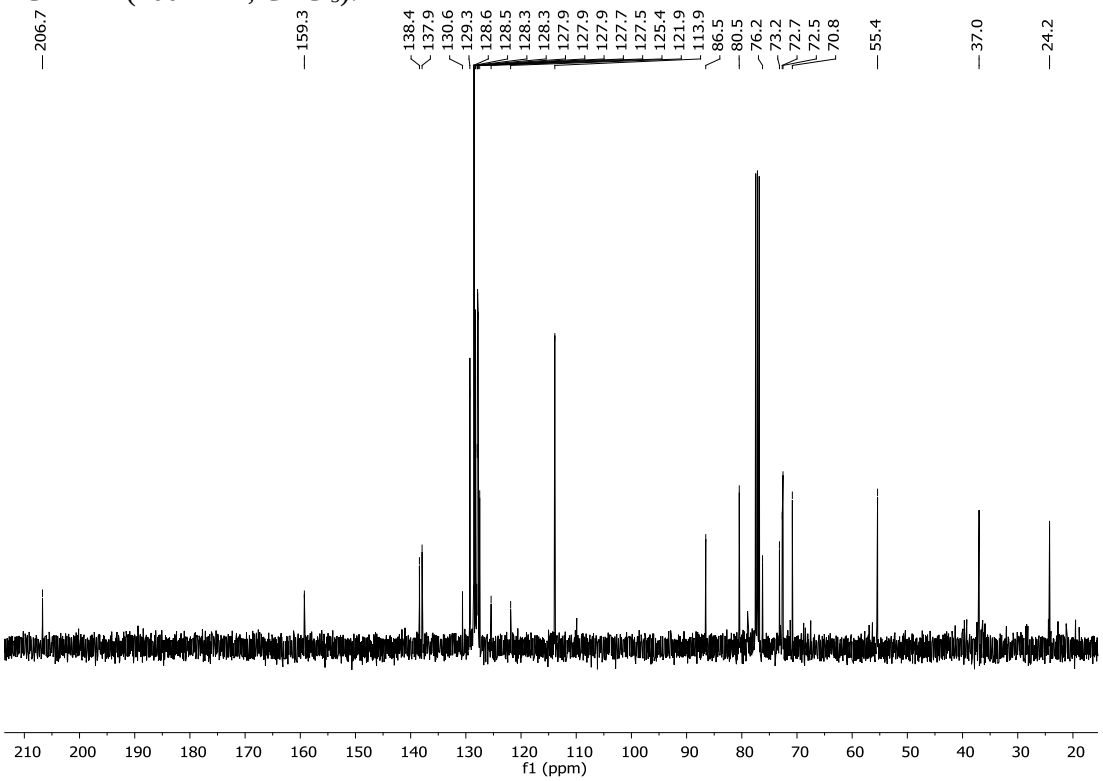


(2*S*, 3*R*, 4*S*, 5*S*)- 2,3,4-Tri-*O*-benzyl-5-*p*-methoxybenzyl-cycloheptanon (15S):

^{1}H-NMR (400 MHz, $CDCl_3$):

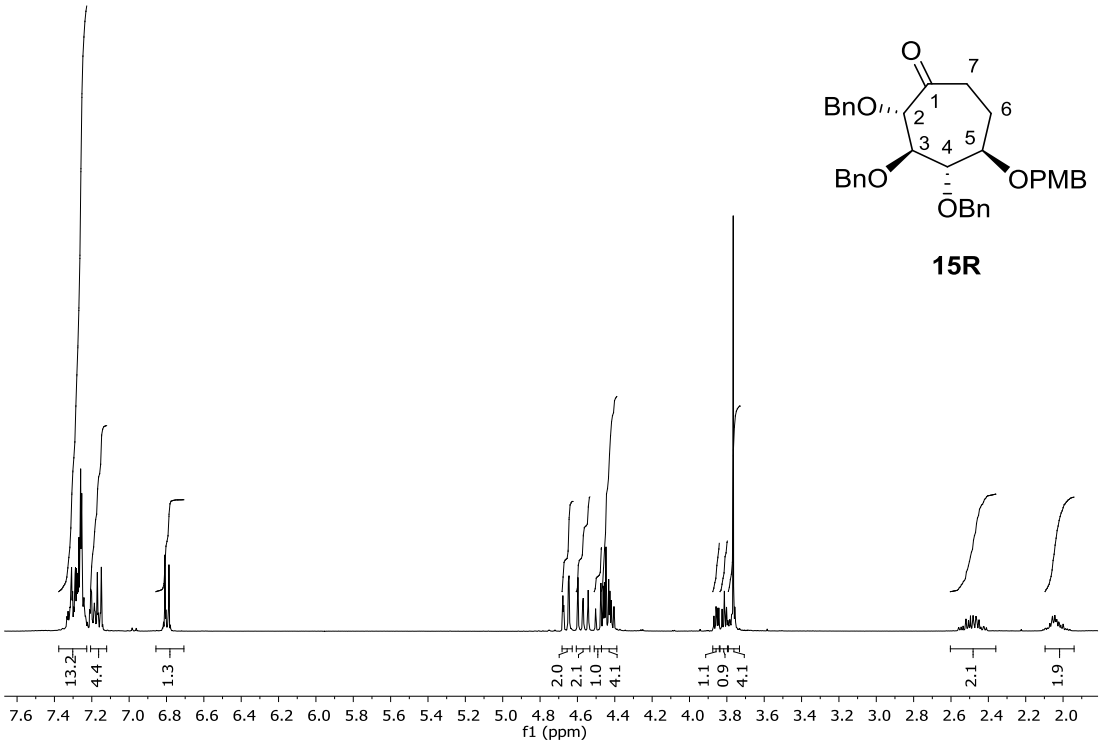


^{13}C-NMR (100 MHz, $CDCl_3$):

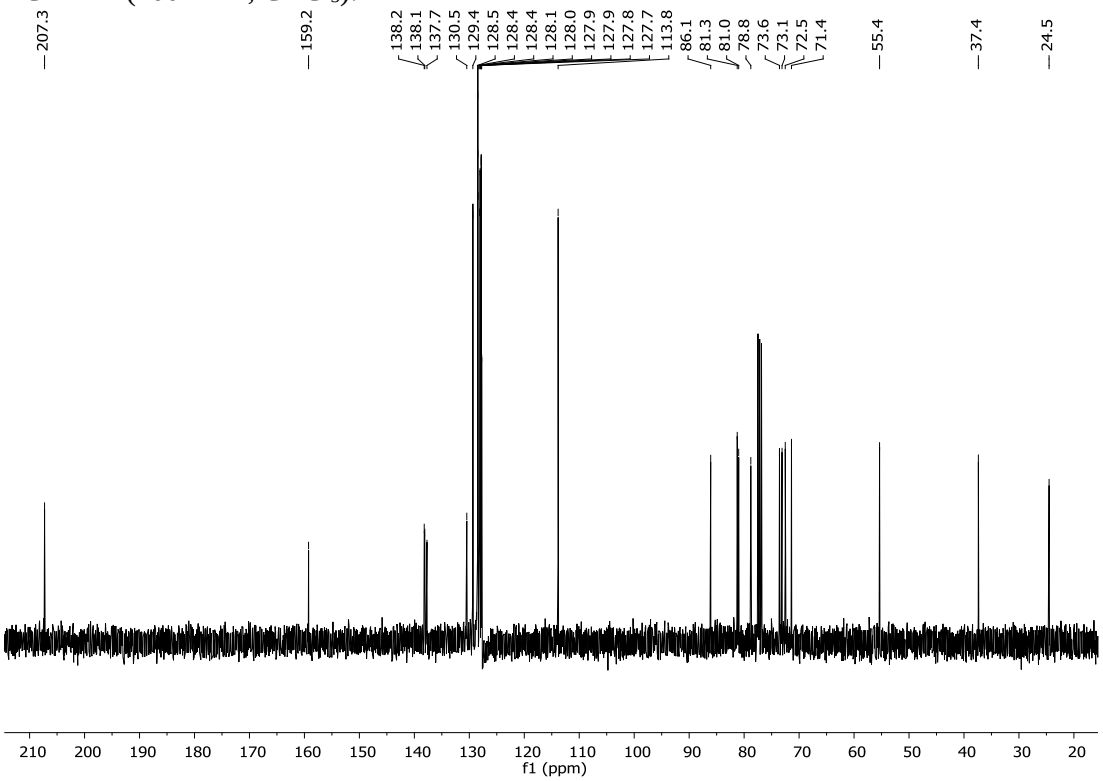


(2*S*, 3*R*, 4*S*, 5*R*)-2,3,4-Tri-*O*-benzyl-5-*p*-methoxybenzylcycloheptanon (15R):

^{1}H-NMR (400 MHz, $CDCl_3$):

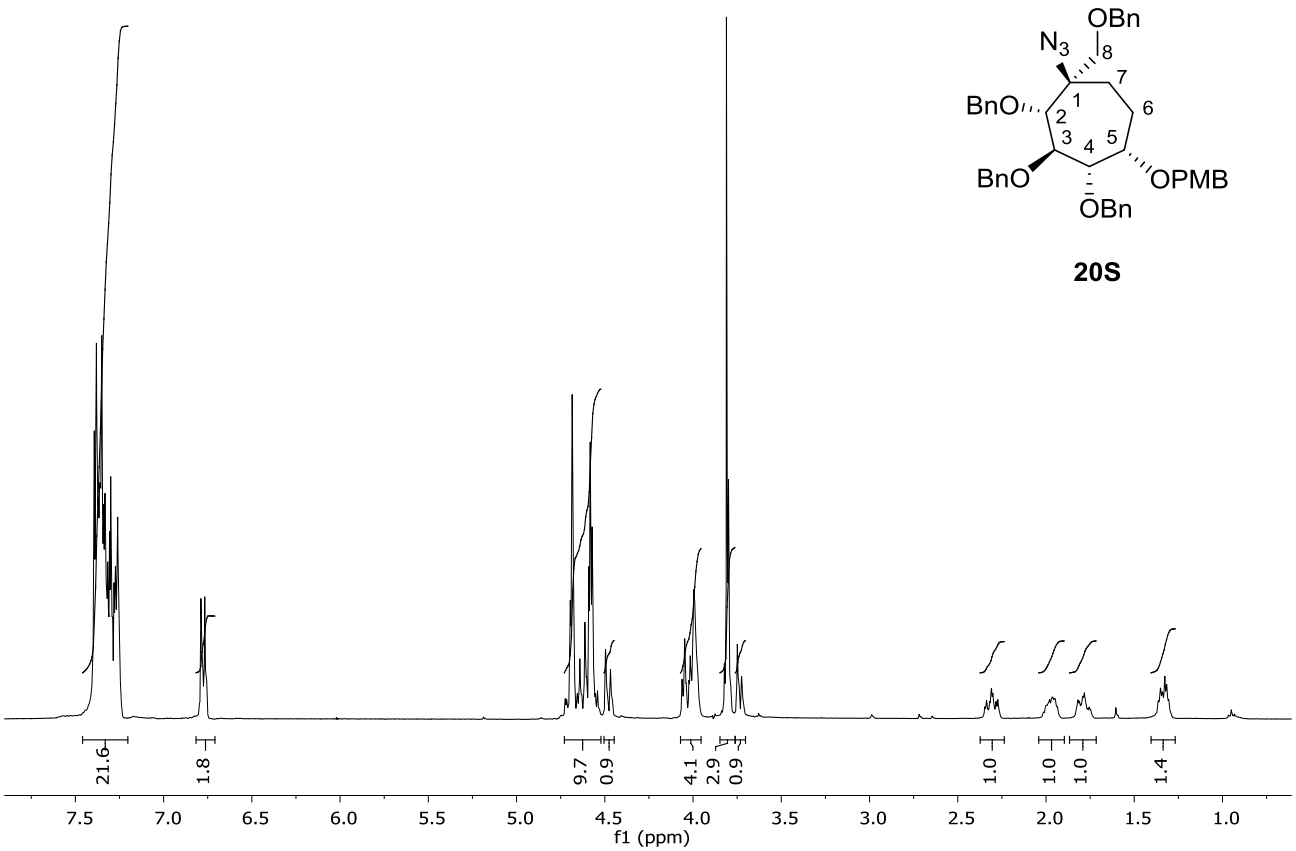


^{13}C-NMR (100 MHz, $CDCl_3$):



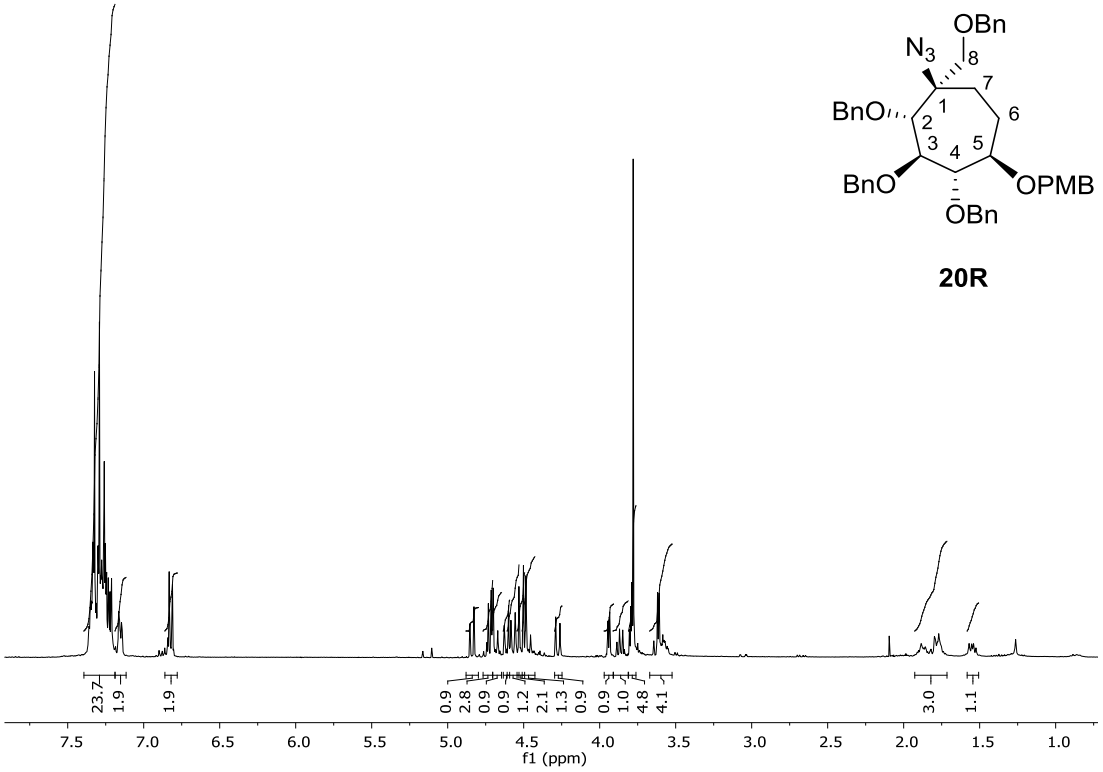
(1*R*, 2*R*, 3*S*, 4*S*, 5*S*)-1-Azido-2,3,4-tri-*O*-benzyl-1-(benzyloxy)methyl-5-*O*-*p*-methoxybenzyl-cycloheptane (20S):

^{1}H-NMR (400 MHz, $CDCl_3$):

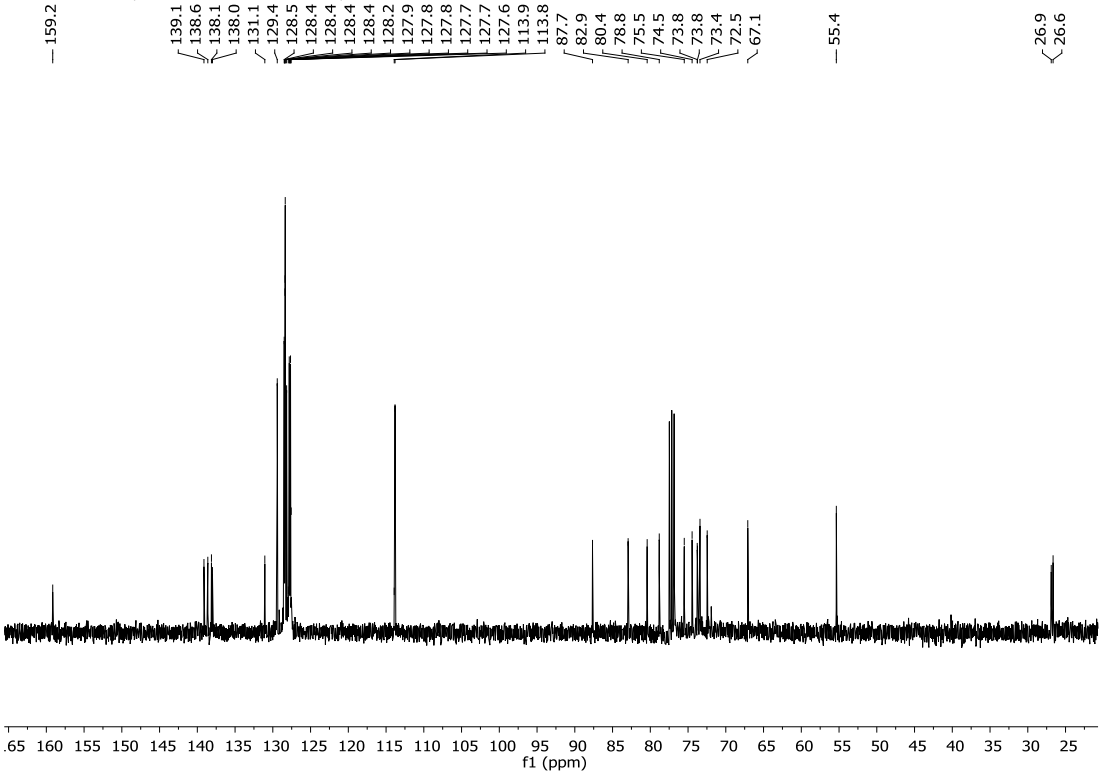


(1*R*, 2*R*, 3*S*, 4*S*, 5*R*)-1-Azido-2,3,4-tri-*O*-benzyl-1-(benzyloxy)methyl-5-*O*-*p*-methoxybenzyl-cycloheptane (20R):

^{1}H-NMR (400 MHz, $CDCl_3$):

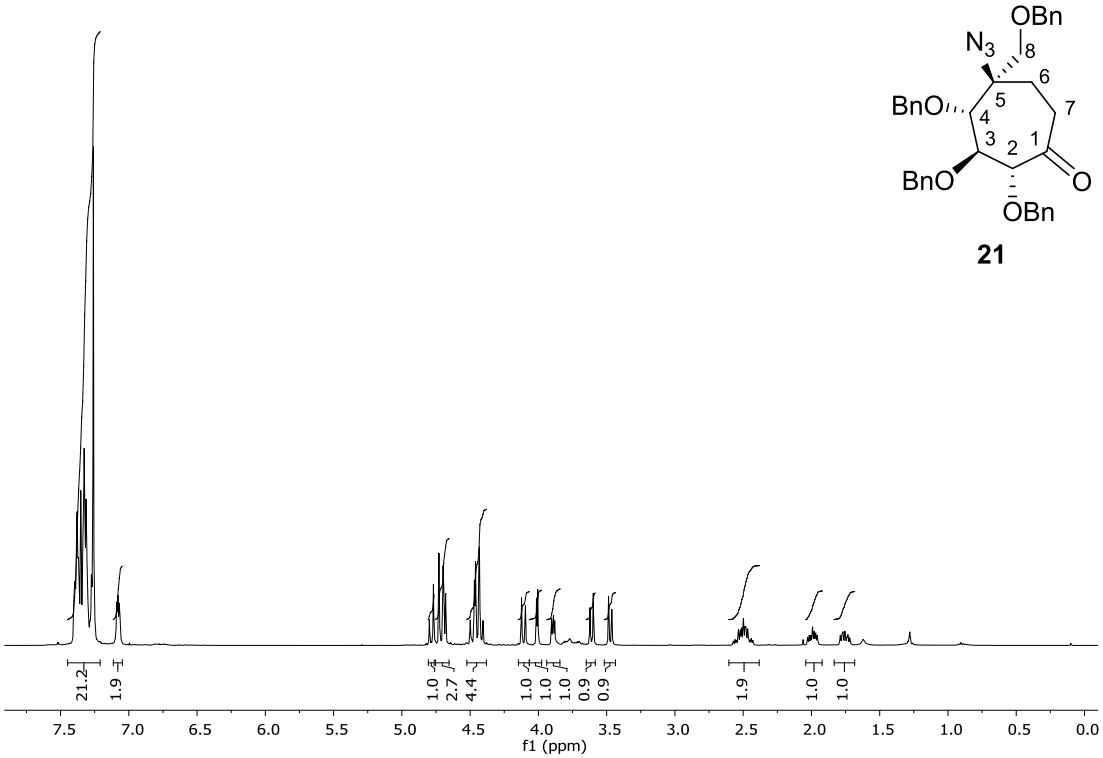


^{13}C-NMR (100 MHz, $CDCl_3$):

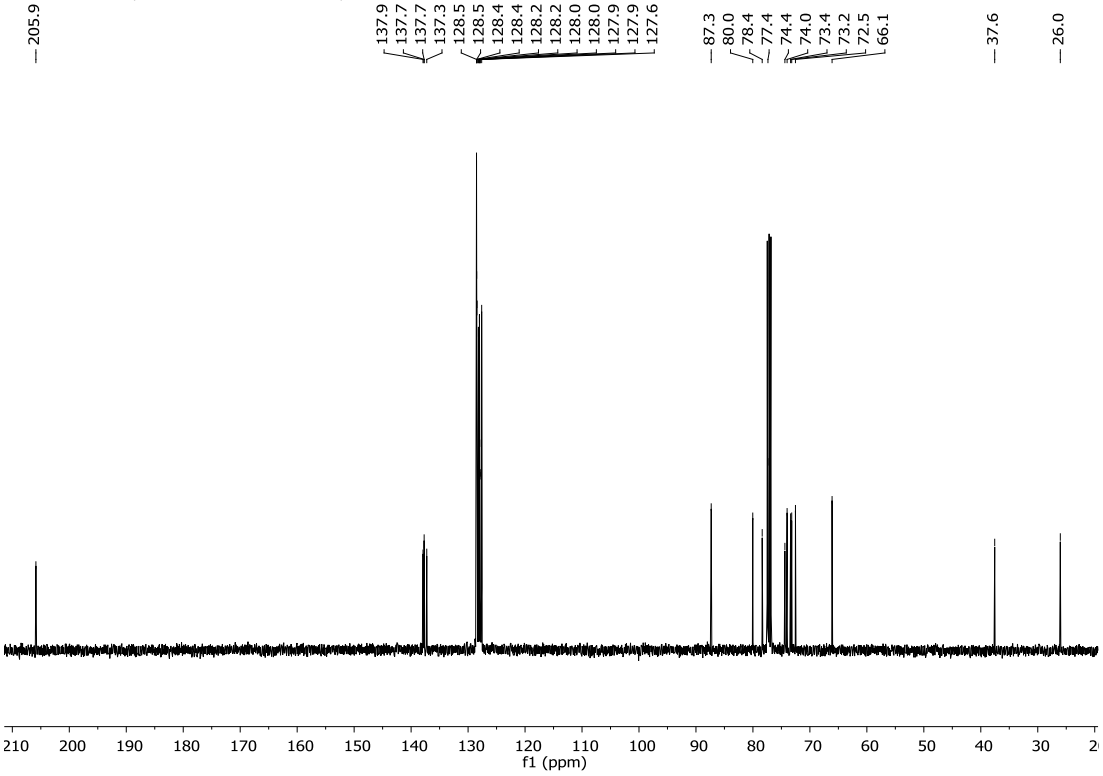


(2*R*,3*S*,4*R*,5*R*)-5-Azido-2,3,4-tri-*O*-benzyl-5-(benzyloxy)methyl-cycloheptanon (21):

^{1}H-NMR (400 MHz, $CDCl_3$):

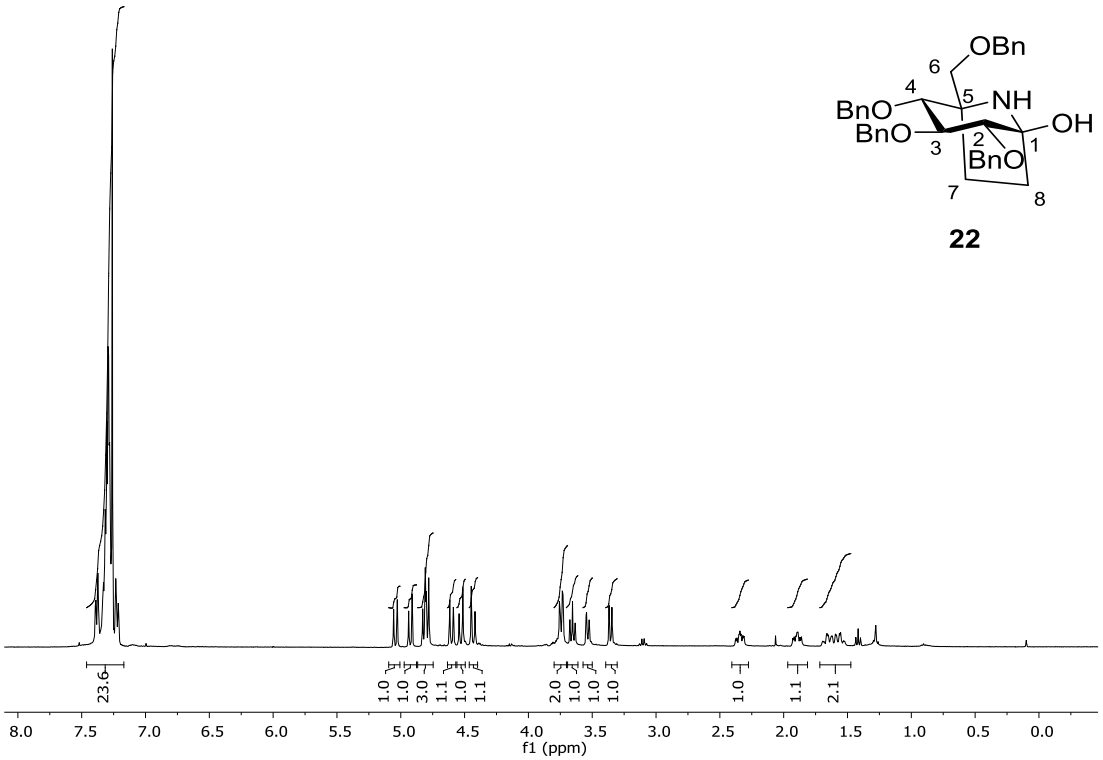


^{13}C-NMR (100 MHz, $CDCl_3$):

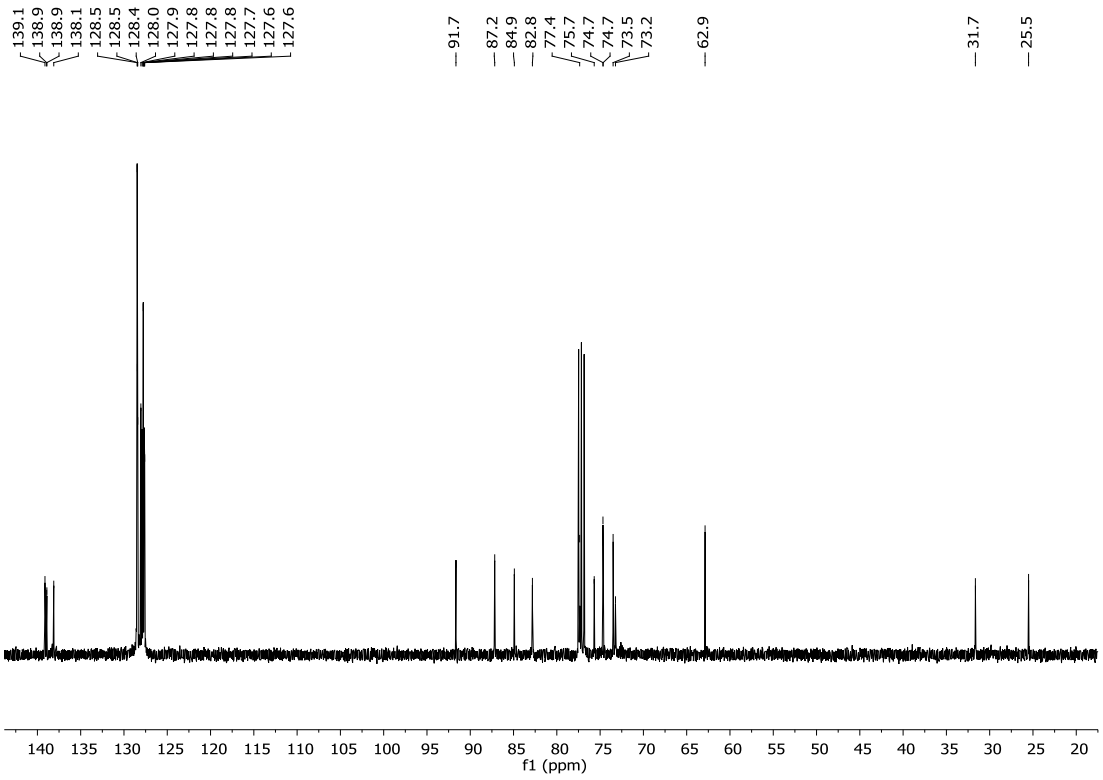


(1S,2R,3S,4R,5R)-2,3,4-tris(benzyloxy)-5-((benzyloxy)methyl)-8-azabicyclo[3.2.1]octan-1-ol (22)

^{1}H-NMR (400 MHz, $CDCl_3$):

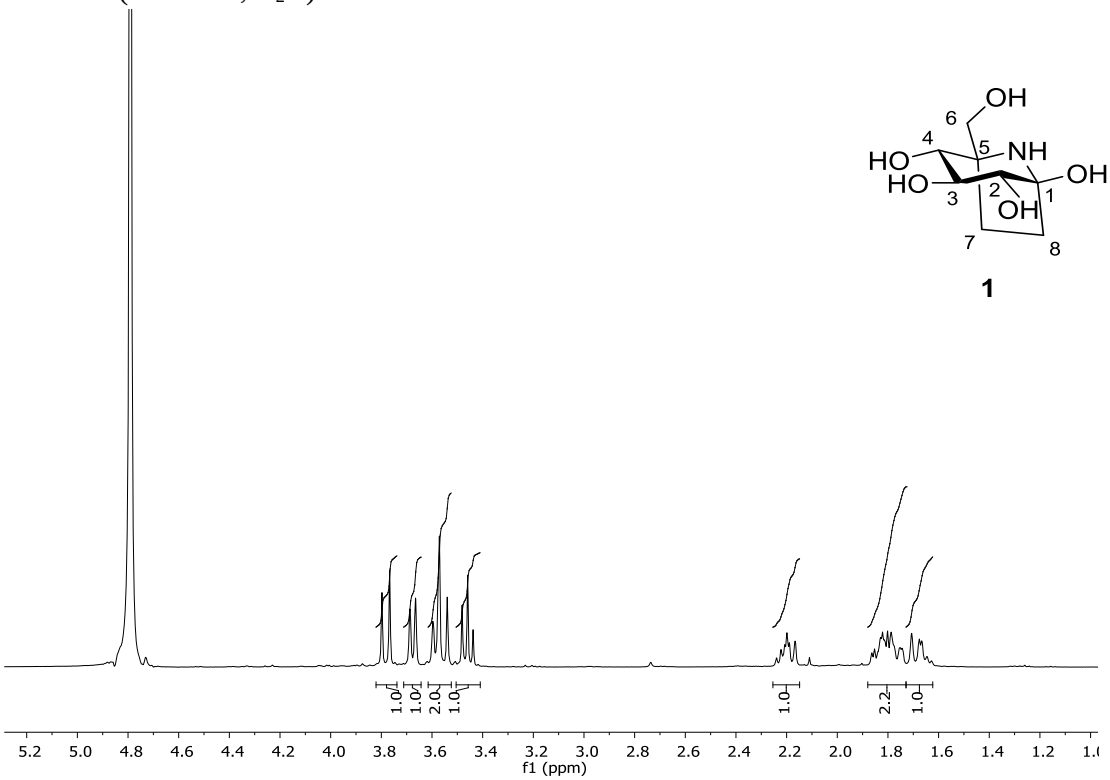


^{13}C-NMR (100 MHz, $CDCl_3$):



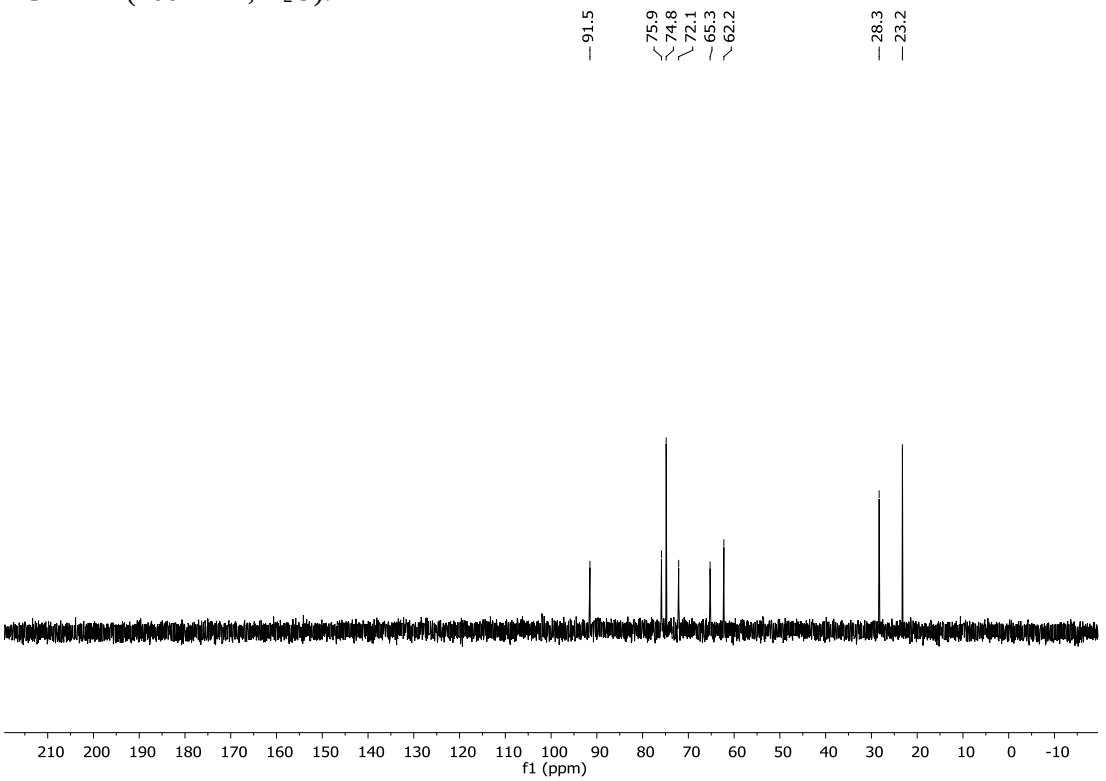
Nojiristegine (1):

^{1}H-NMR (400 MHz, D_2O):



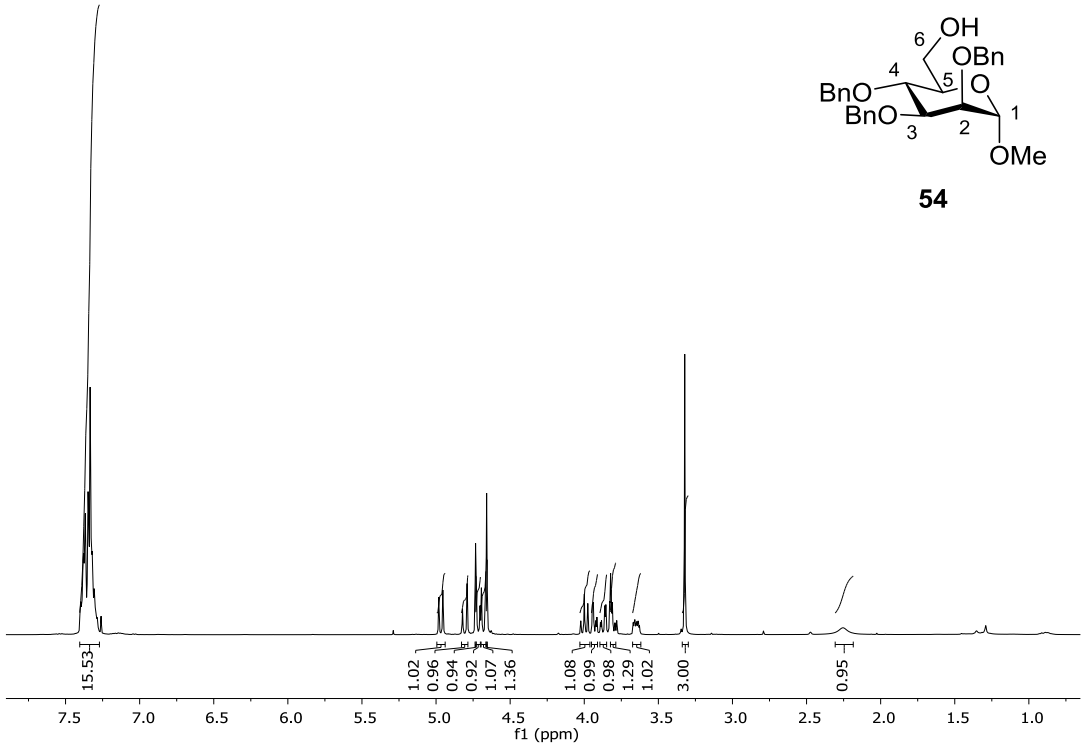
1

^{13}C-NMR (100 MHz, D_2O):



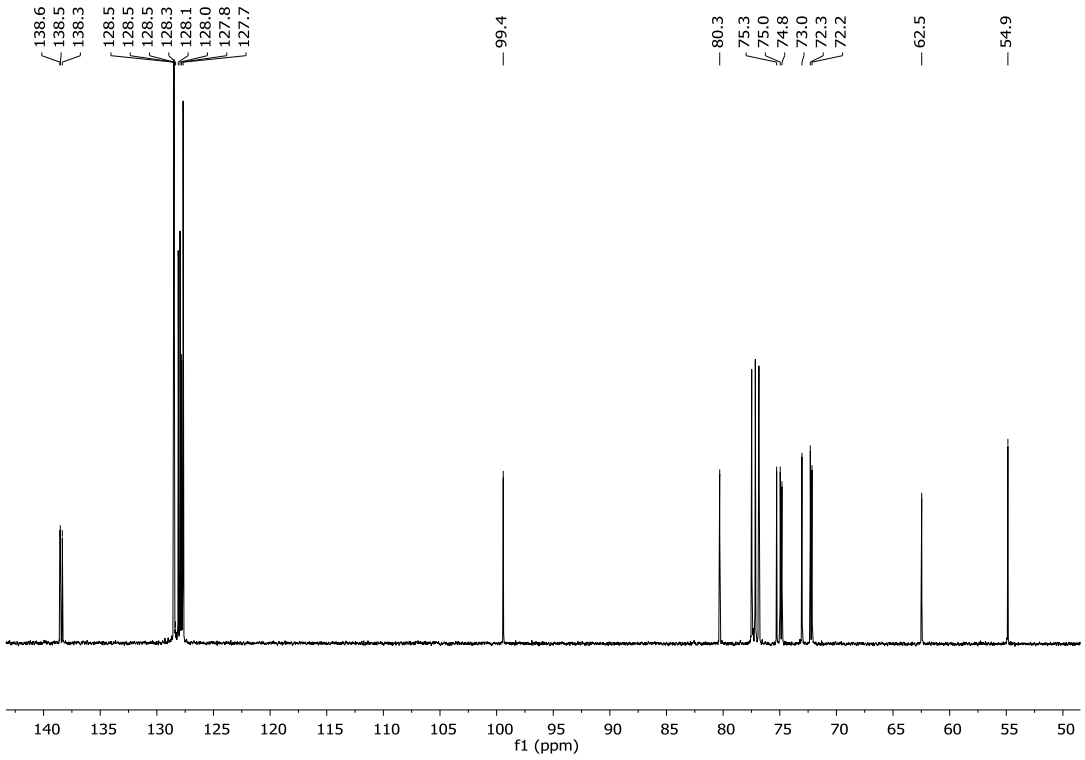
Methyl 2,3,4-tri-*O*-benzyl-6-hydroxy-α-D-mannopyranoside (54):

^{1}H-NMR (400 MHz, $CDCl_3$):



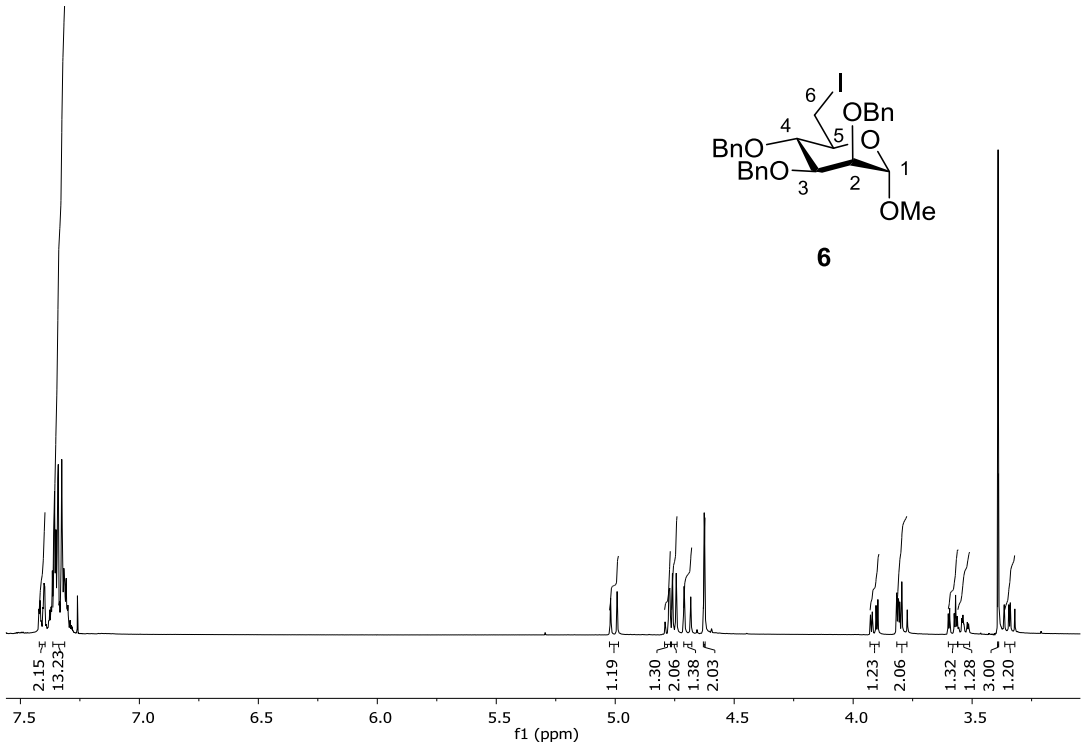
54

^{13}C-NMR (100 MHz, $CDCl_3$):



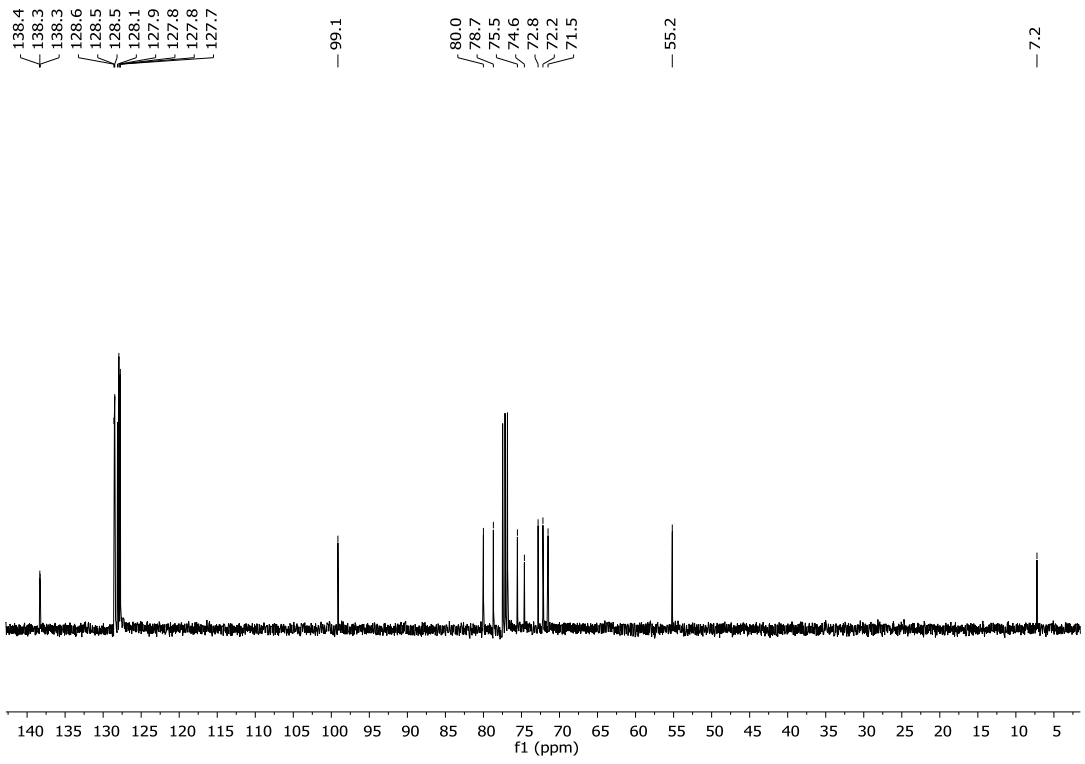
Methyl 2,3,4-tri-*O*-benzyl-6-deoxy-6-iodo-α-D-mannopyranoside (6):

^{1}H-NMR (400 MHz, $CDCl_3$):



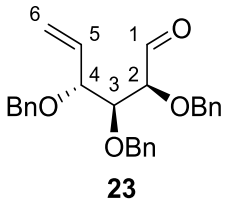
6

^{13}C-NMR (100 MHz, $CDCl_3$):



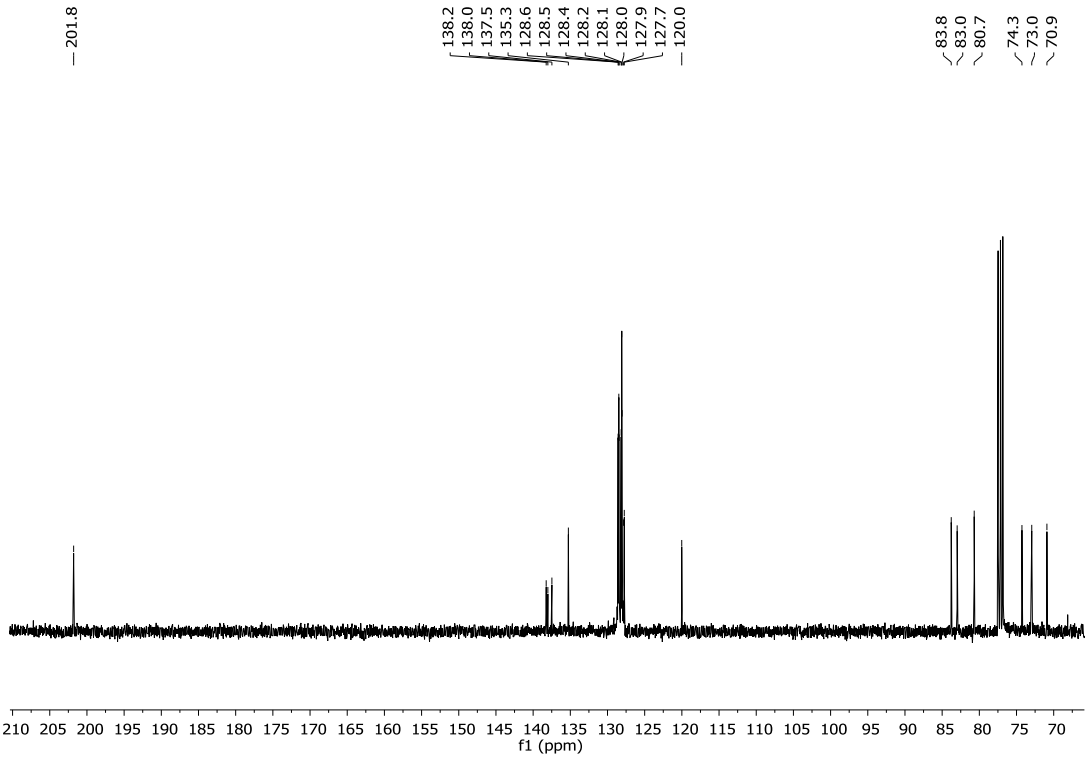
(2*S*, 3*S*, 4*R*)-2,3,4-Tri-*O*-benzylhex-5-enal (23):

^{1}H-NMR (400 MHz, $CDCl_3$):



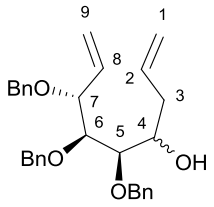
23

^{13}C-NMR (100 MHz, $CDCl_3$):



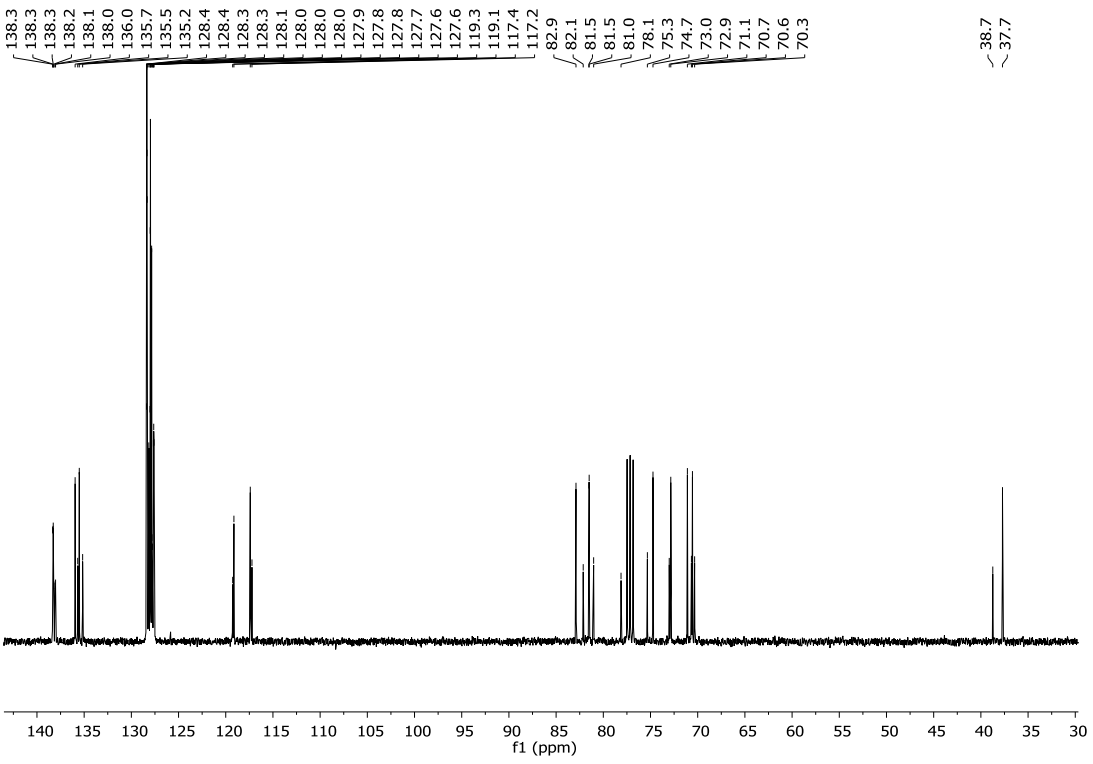
(4*R*/*S*, 5*R*, 6*S*, 7*R*)-5,6,7-Tri-*O*-benzyl-1,8-nonadiene-4-ol (24):

^{1}H-NMR (400 MHz, $CDCl_3$):



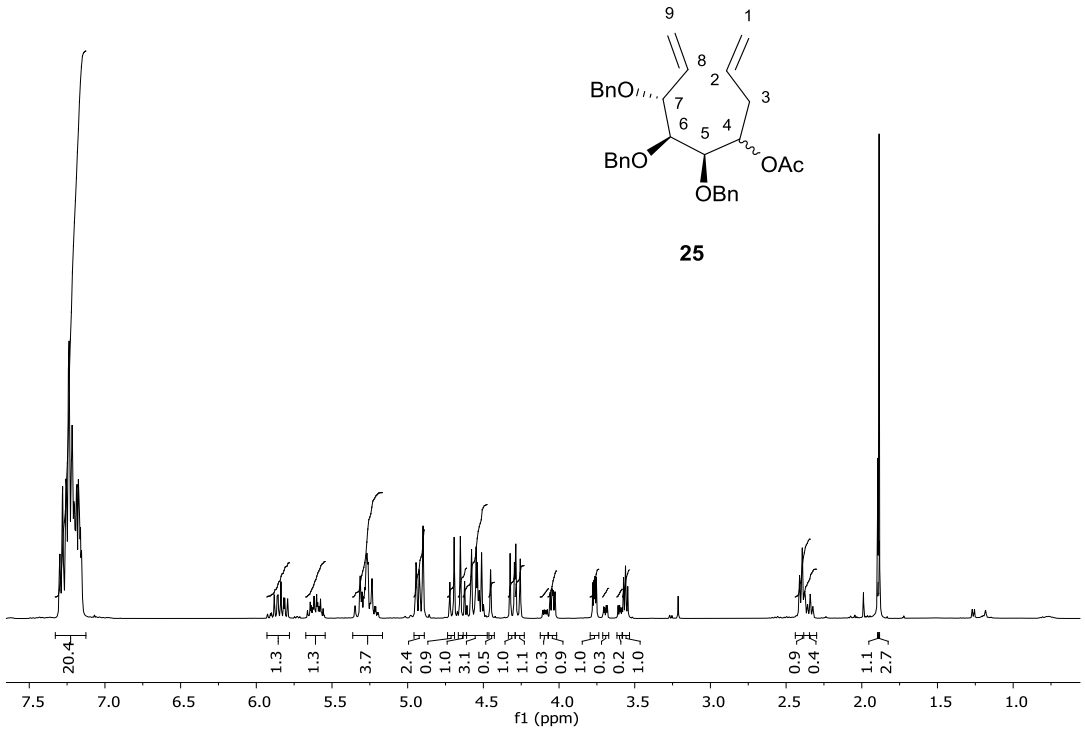
24

^{13}C-NMR (100 MHz, $CDCl_3$):

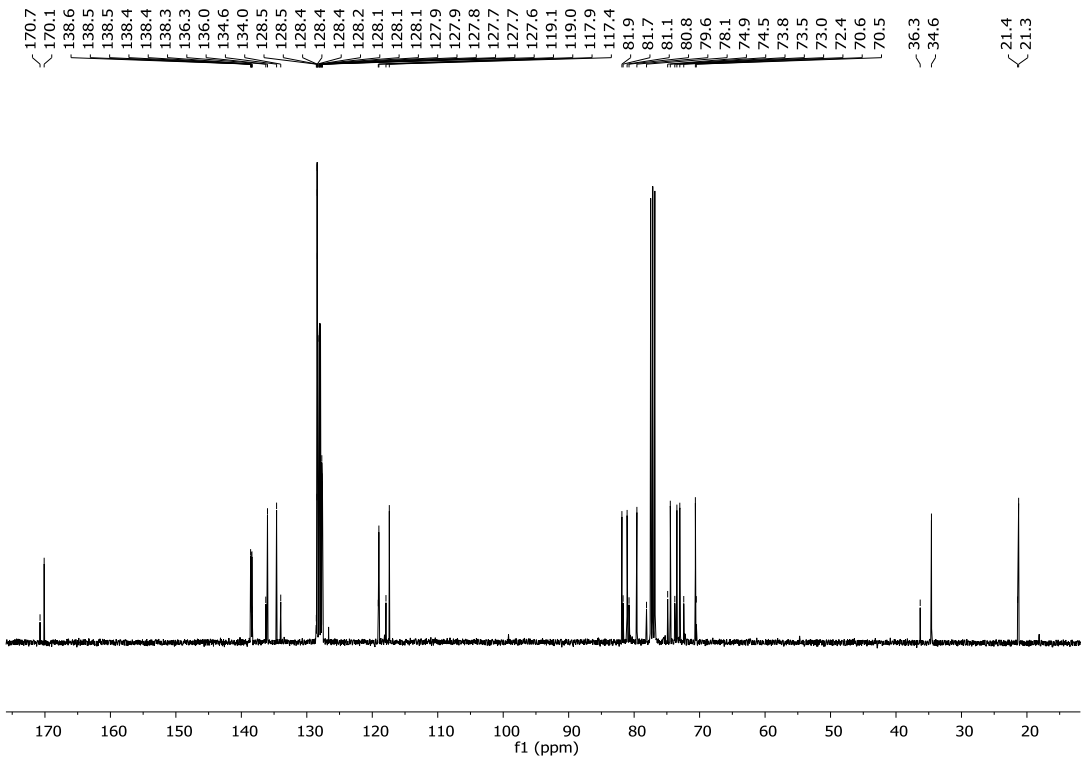


(4*R*/*S*,5*R*,6*S*,7*R*)-5,6,7-tri-*O*-benzyl1,8-nonadiene-4-acetate (25):

^{1}H-NMR (400 MHz, $CDCl_3$):

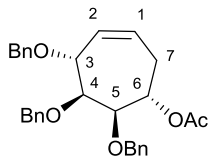


^{13}C-NMR (100 MHz, $CDCl_3$):

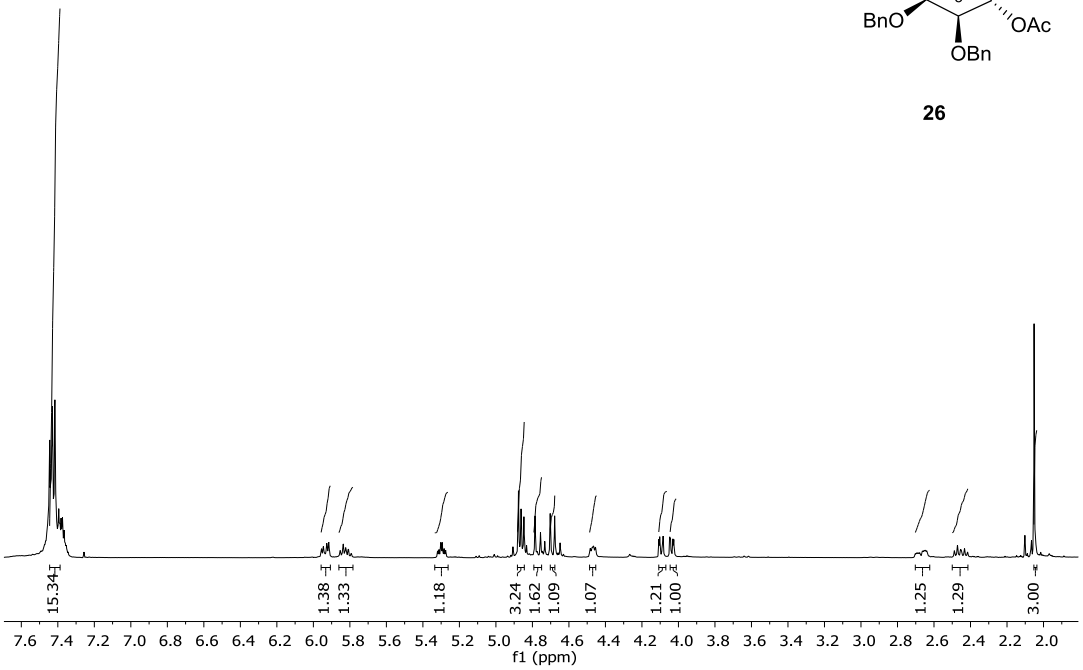


(3*R*,4*S*,5*R*,6*S*)- 6-*O*-Acetyl-3,4,5-tri-*O*-benzyl-cycloheptene (26):

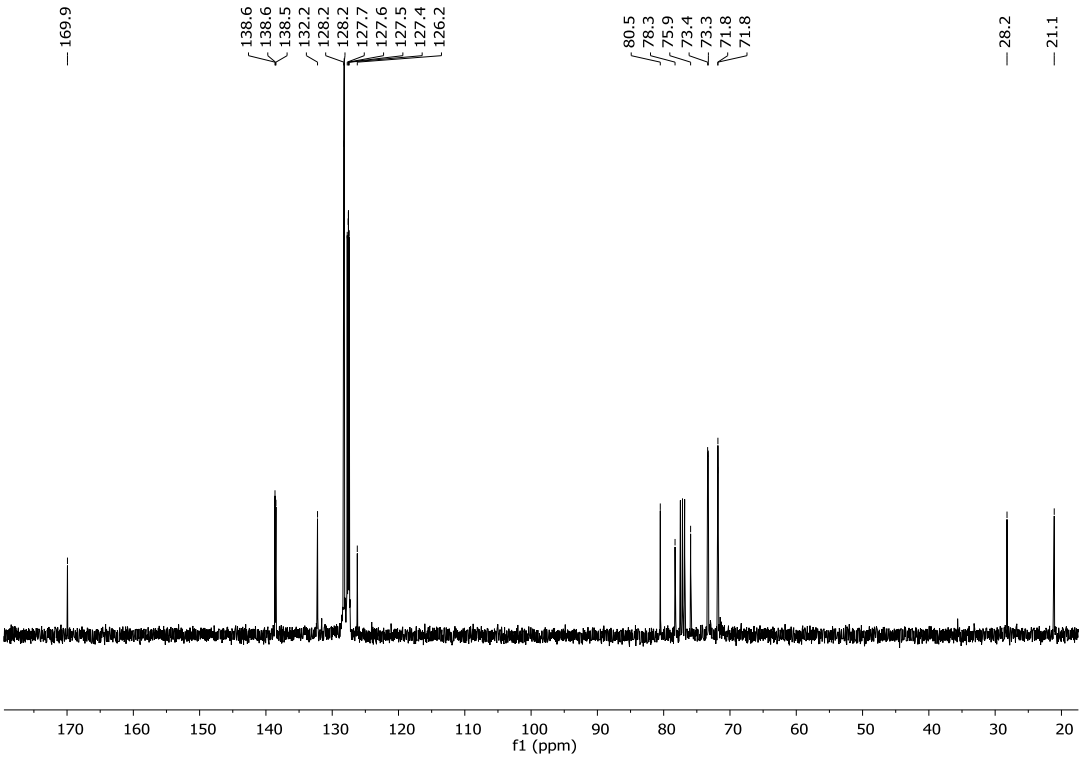
^{1}H-NMR (400 MHz, $CDCl_3$):



26

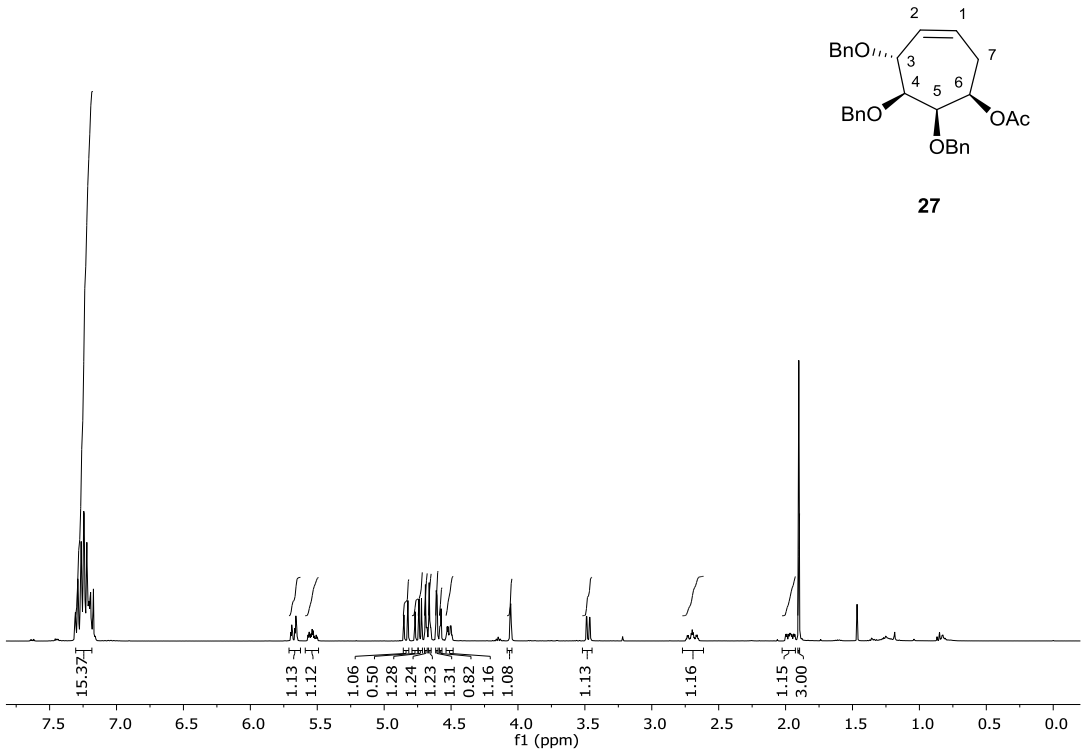


^{13}C-NMR (100 MHz, $CDCl_3$):

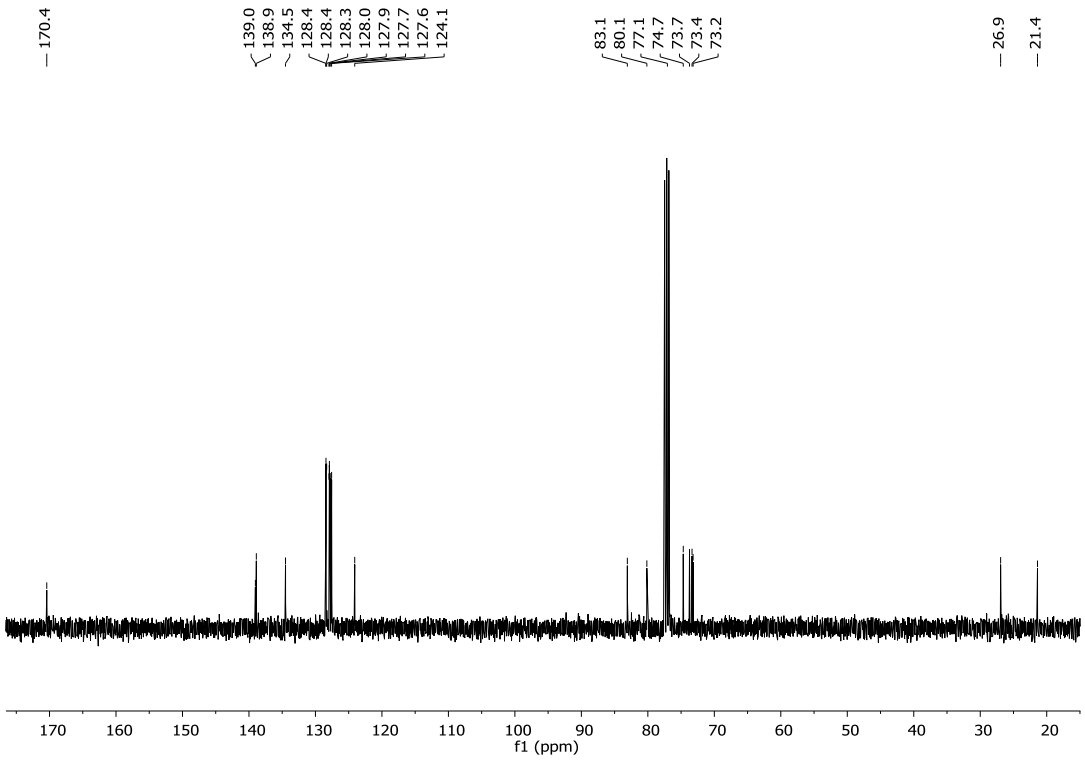


(3*R*,4*S*,5*R*,6*R*)- 6-*O*-Acetyl-3,4,5-tri-*O*-benzyl-cycloheptene (27):

^{1}H-NMR (400 MHz, $CDCl_3$):

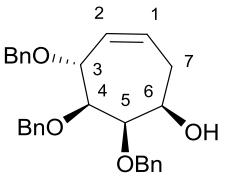


^{13}C-NMR (100 MHz, $CDCl_3$):

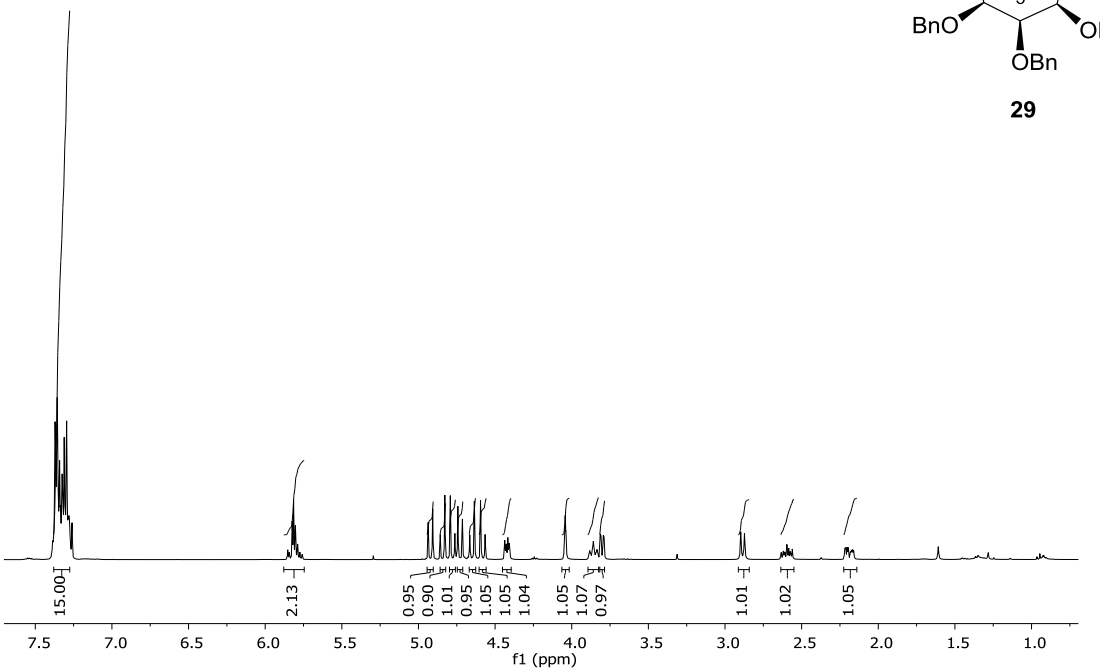


(3R,4S,5R,6R)-3,4,5-Tri-O-benzyl-6-hydroxy-cycloheptene (29):

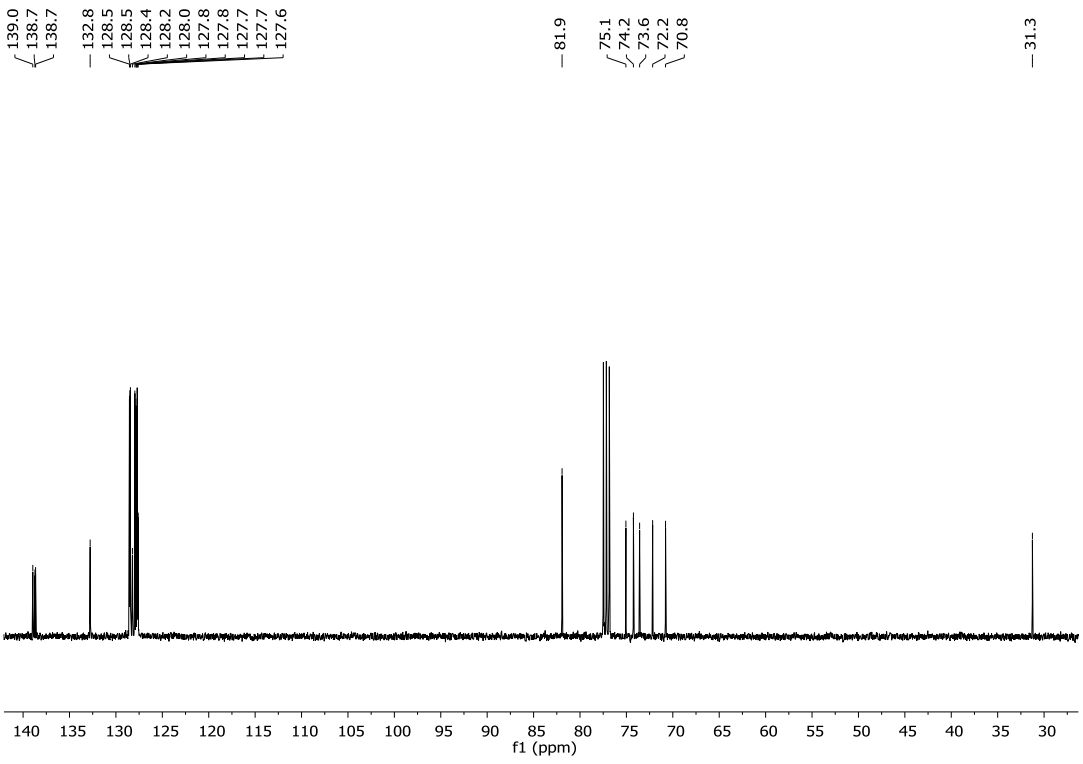
^{1}H-NMR (400 MHz, $CDCl_3$):



29

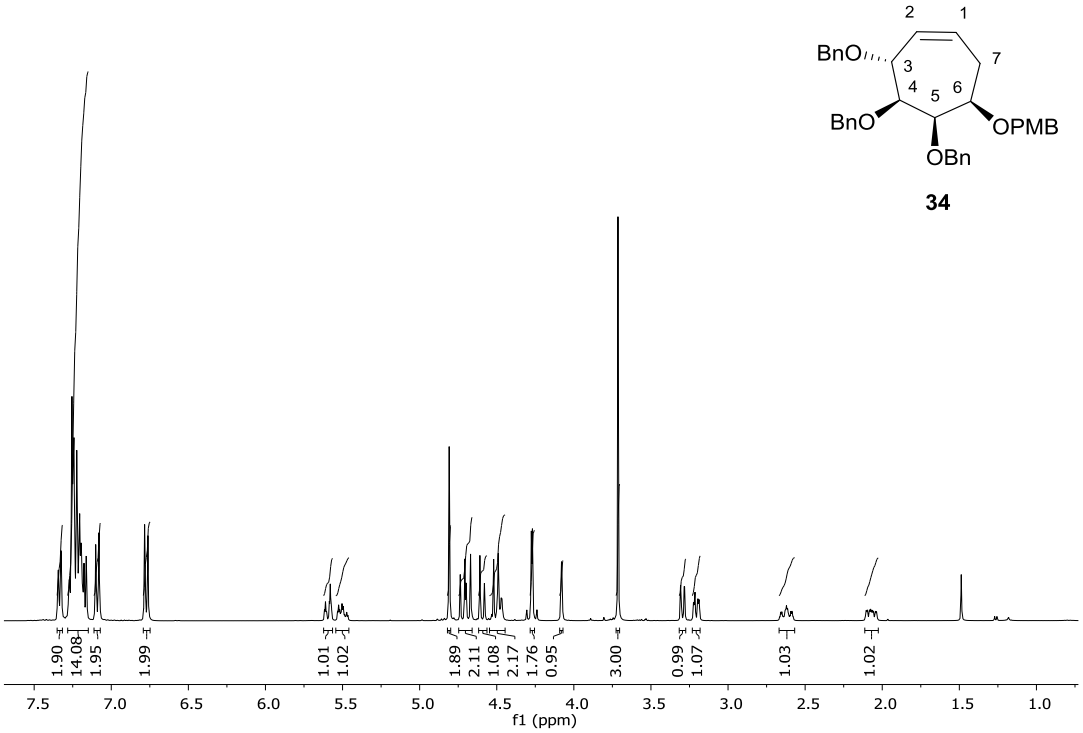


^{13}C-NMR (100 MHz, $CDCl_3$):

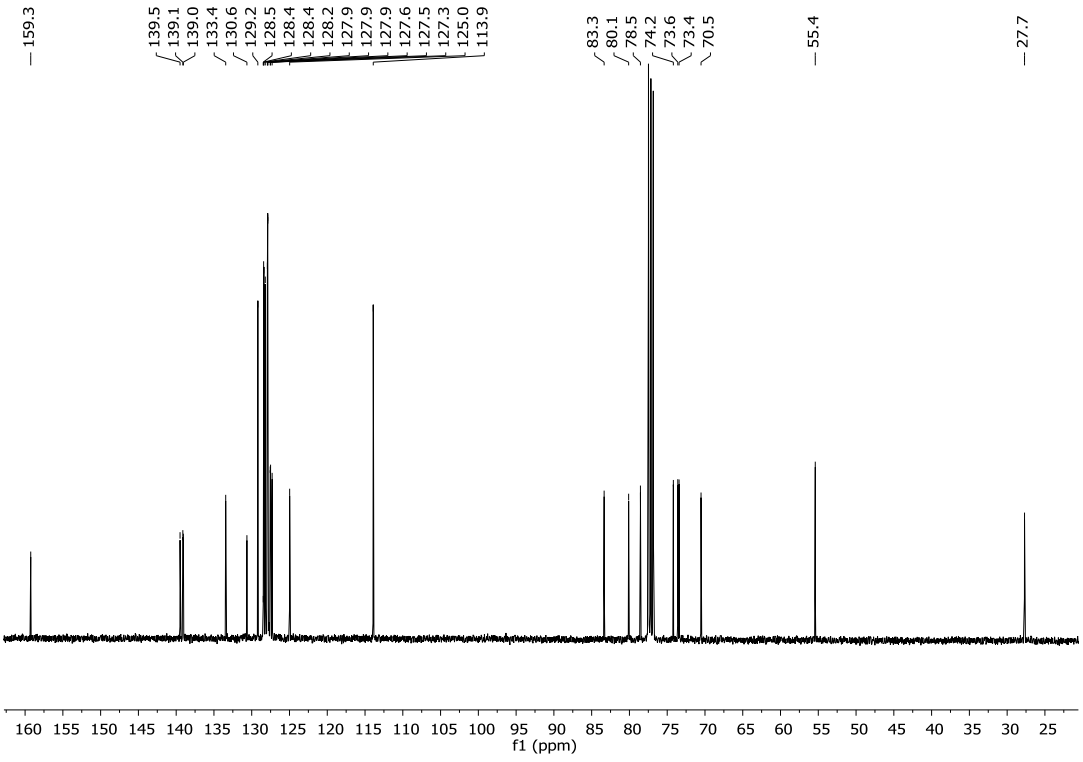


(3*R*,4*S*,5*R*,6*R*)-3,4,5-Tri-*O*-benzyl-6-*O*-p-methoxybenzyl-cycloheptene (34):

^{1}H-NMR (400 MHz, $CDCl_3$):

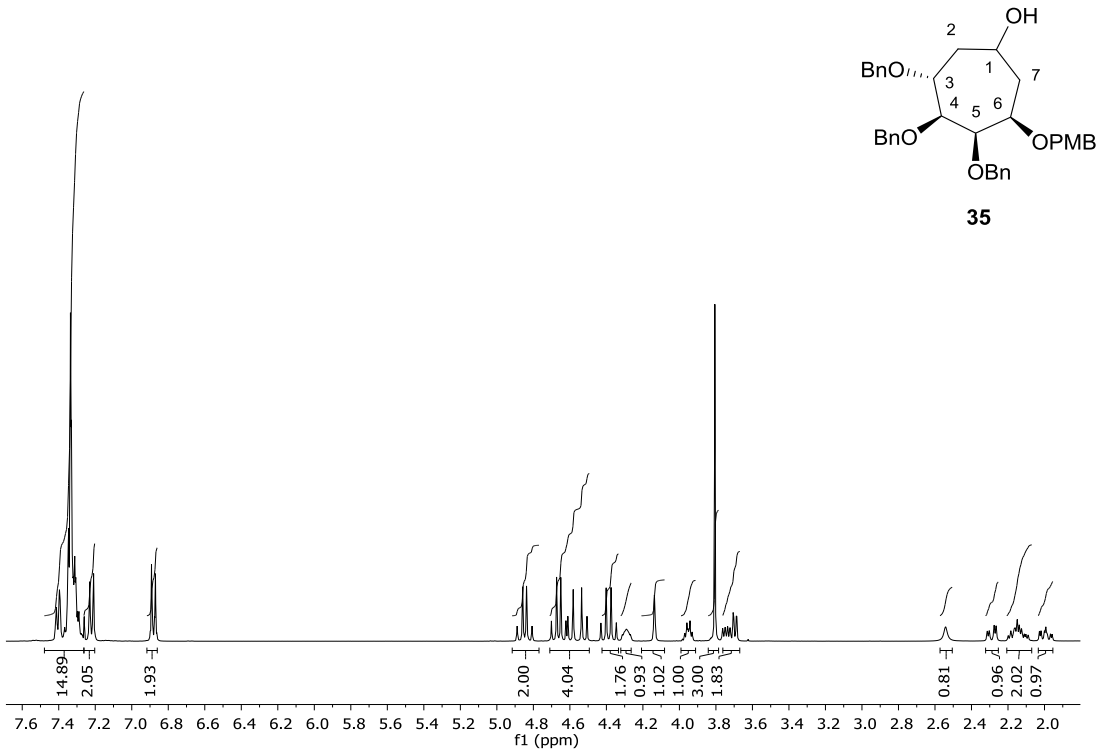


^{13}C-NMR (100 MHz, $CDCl_3$):

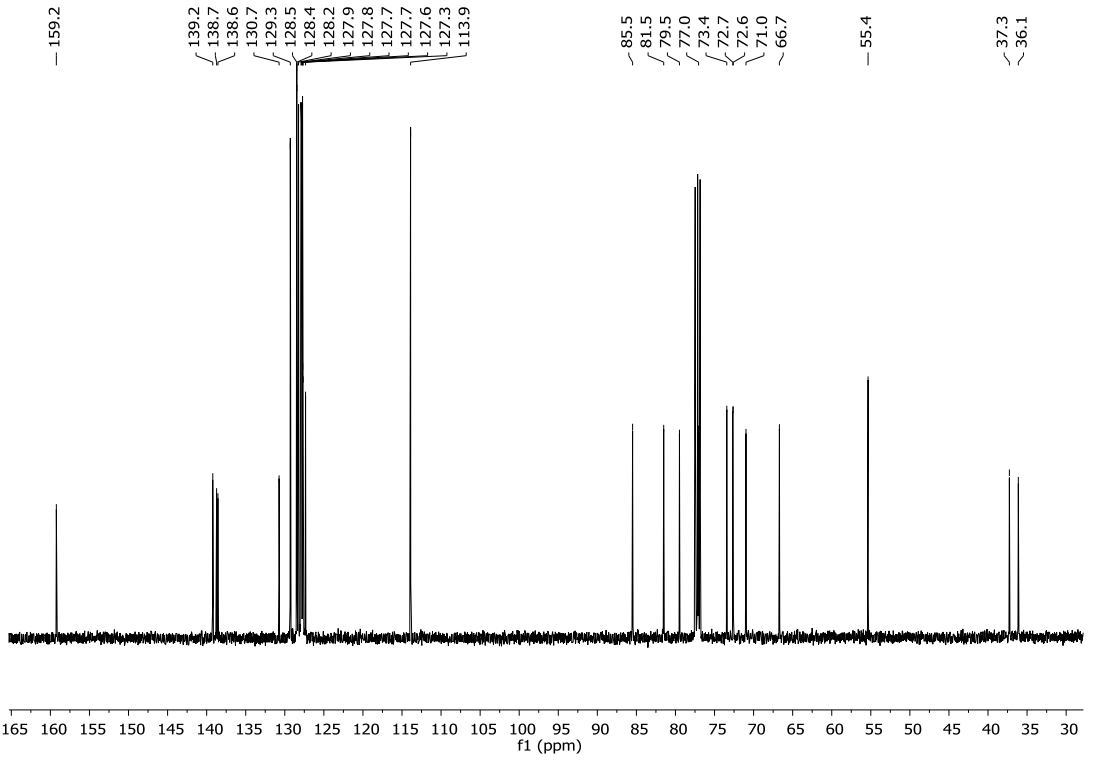


(3*R*,4*S*,5*R*,6*R*)-3,4,5-Tri-*O*-benzyl-6-*O*-p-methoxybenzyl-cycloheptane-1-ol (35):

^{1}H-NMR (400 MHz, $CDCl_3$):

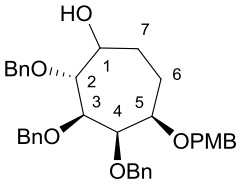


^{13}C-NMR (100 MHz, $CDCl_3$):



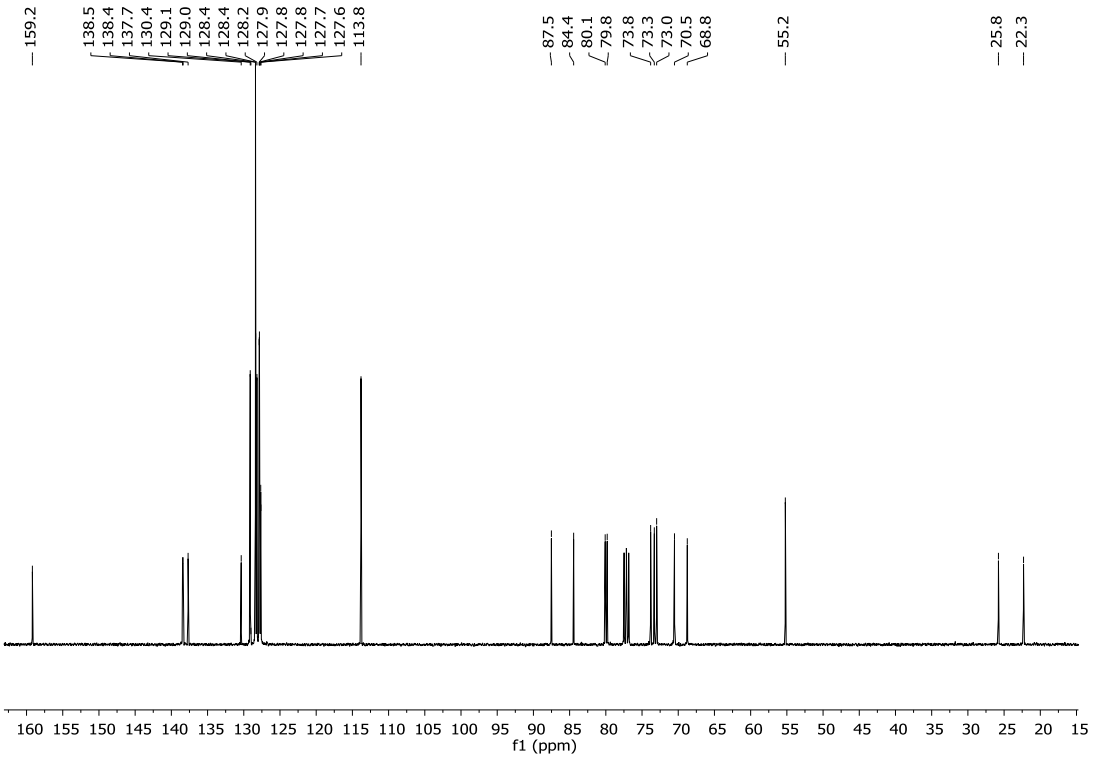
(2*R*,3*S*,4*R*,5*R*)-2,3,4-tri-*O*-benzyl-5-*O*-*p*-methoxybenzyl-cycloheptane-1-ol (36):

^{1}H-NMR (400 MHz, $CDCl_3$):



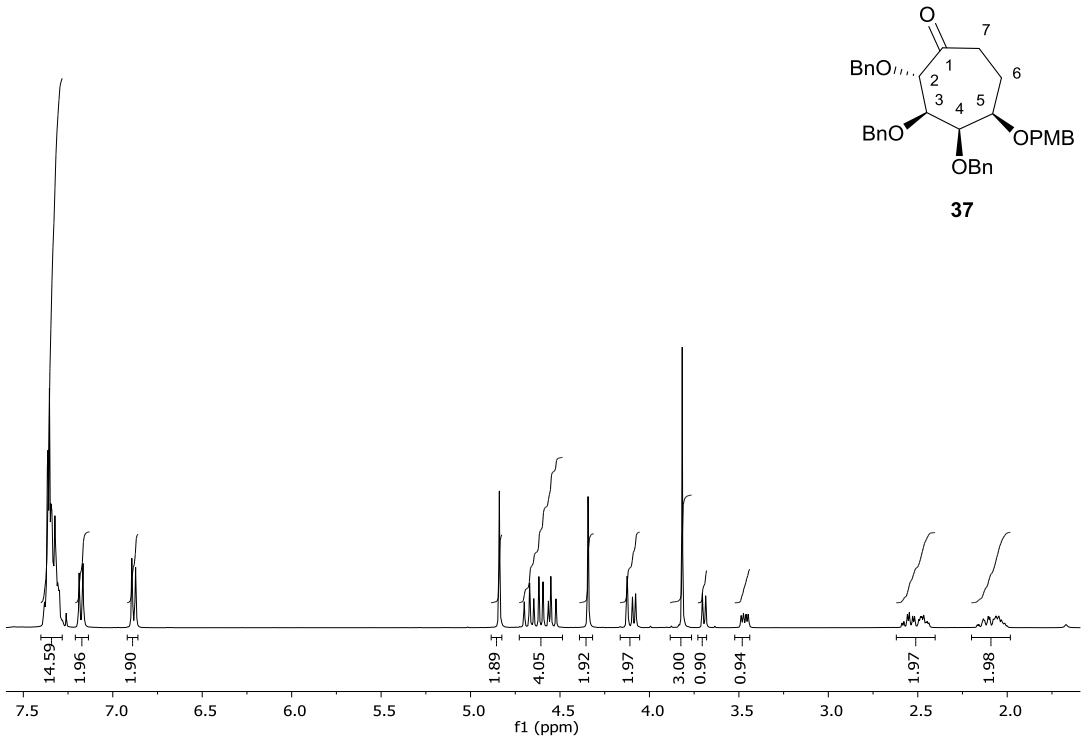
36

^{13}C-NMR (100 MHz, $CDCl_3$):

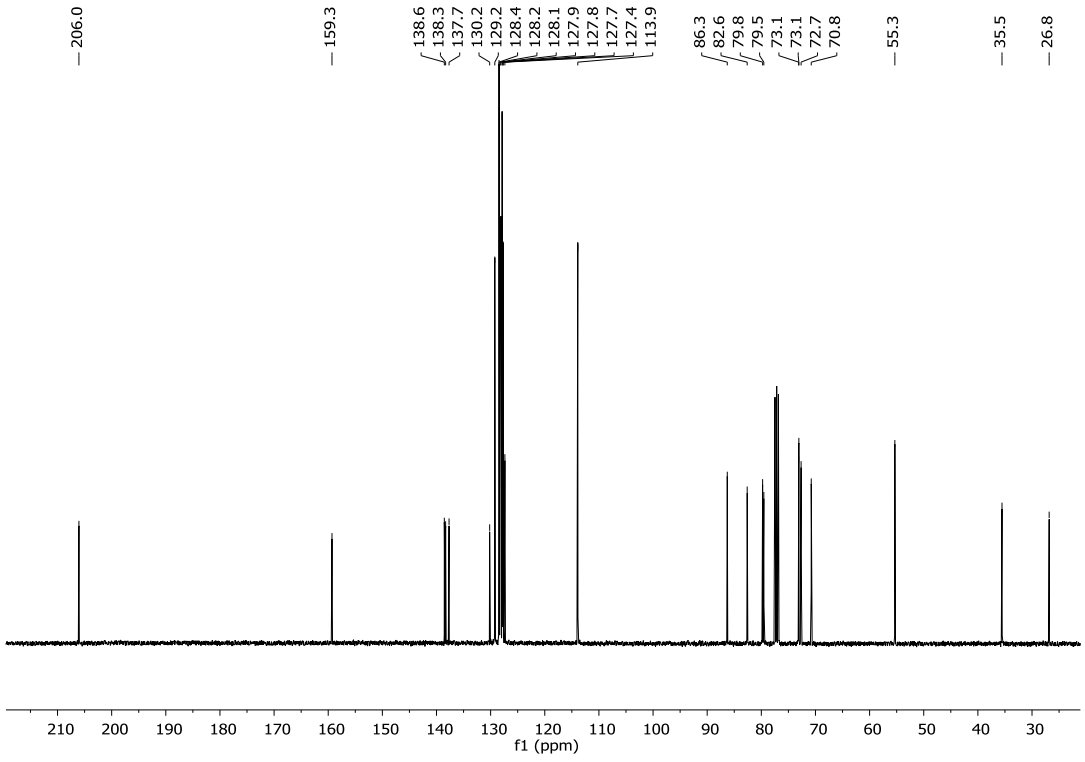


(2*S*,3*R*,4*R*,5*R*)-2,3,4-Tri-*O*-benzyl-5-*p*-methoxybenzyl-cycloheptanone (37):

^{1}H-NMR (400 MHz, $CDCl_3$):

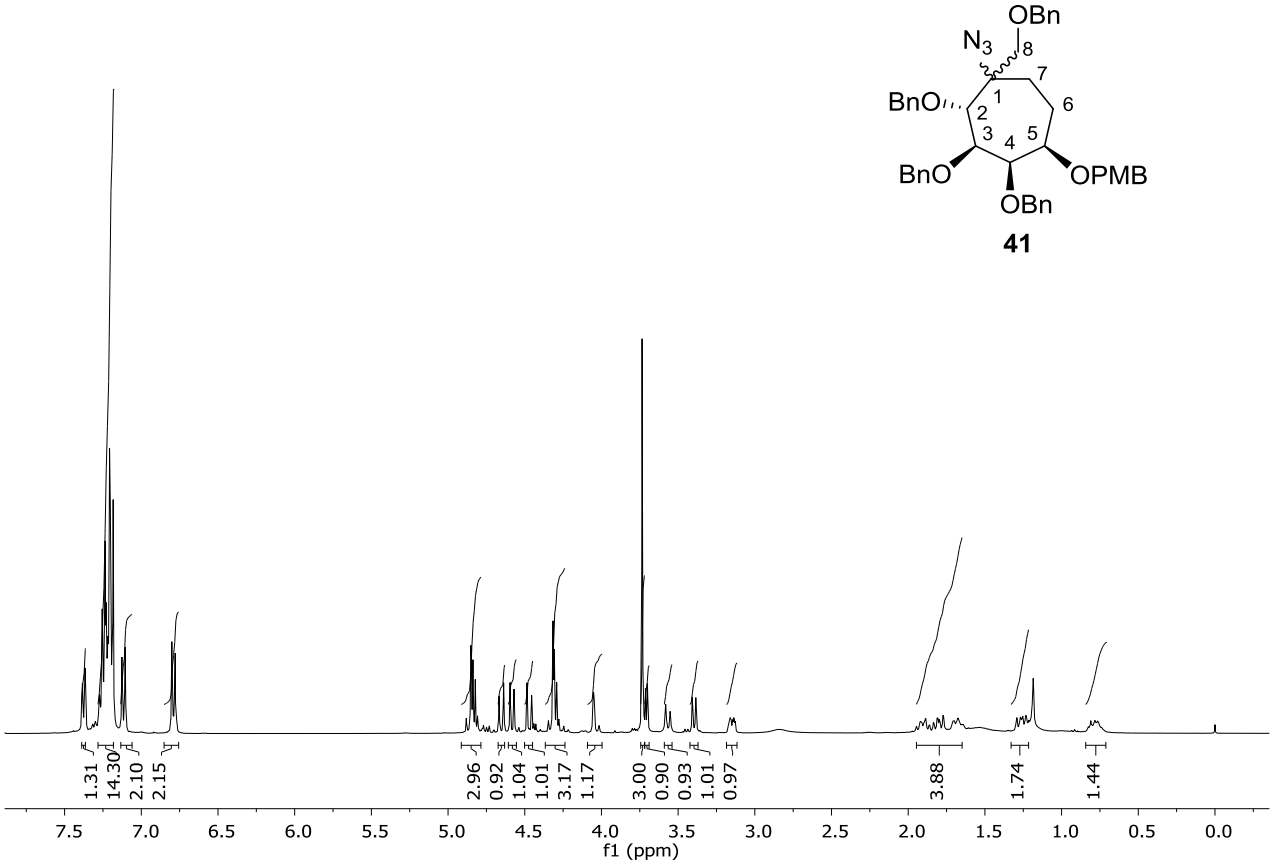


^{13}C-NMR (100 MHz, $CDCl_3$):



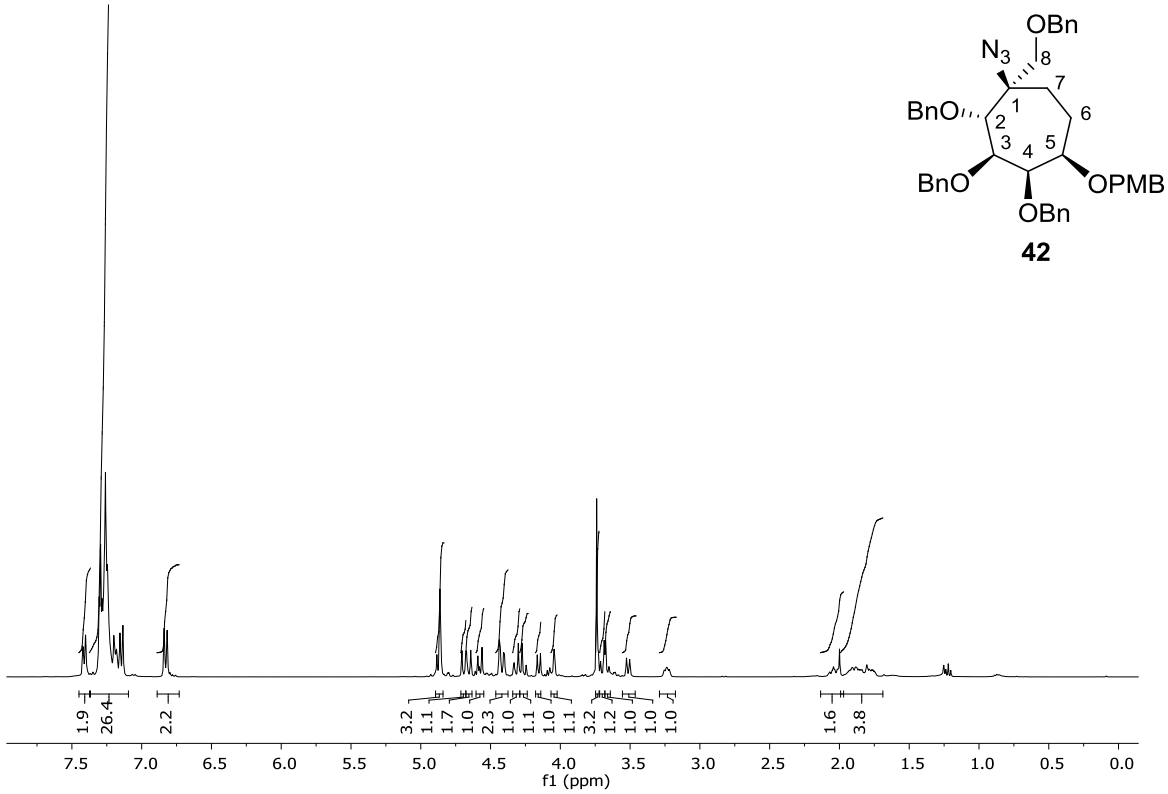
(1*R*/*S*,2*R*,3*S*,4*R*,5*R*)-1-Azido-2,3,4-tri-*O*-benzyl-1-hydroxymethyl-5-*p*-methoxybenzyl-cycloheptane (41)

^{1}H-NMR (400 MHz, $CDCl_3$):

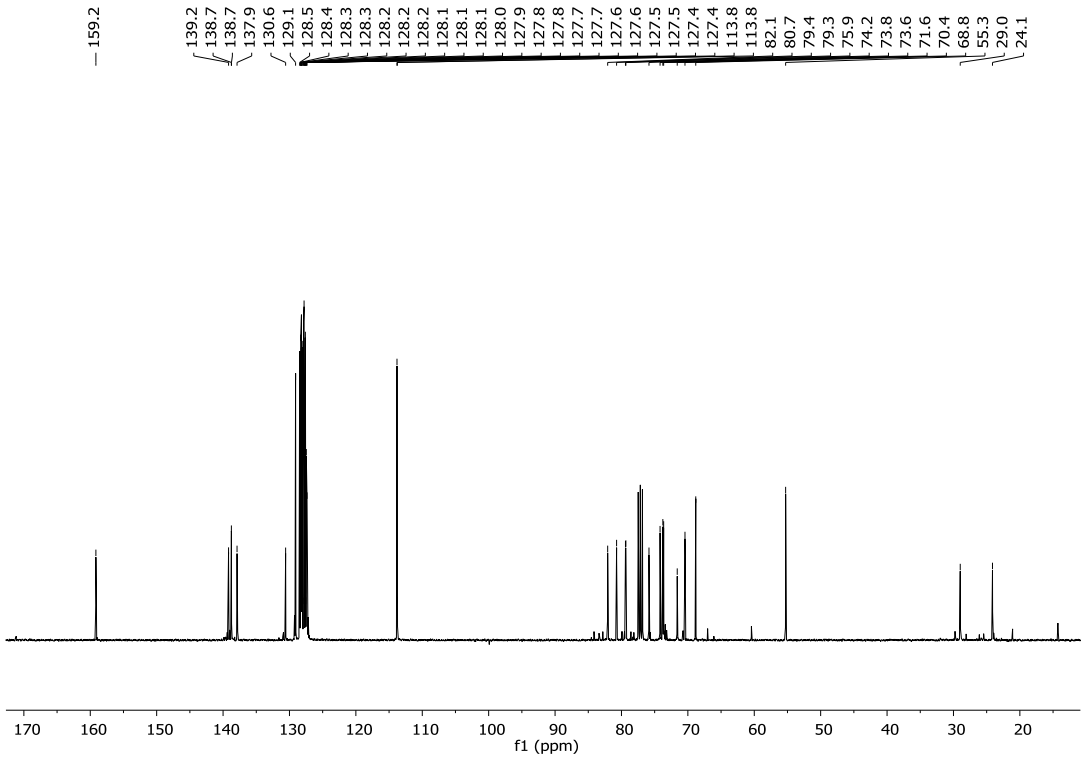


(1*R*,2*R*,3*S*,4*R*,5*R*)-1-azido-2,3,4-tri-*O*-benzyl-1-(benzyloxy)methyl-5-*p*-methoxybenzyl-cycloheptane (42):

^{1}H-NMR (400 MHz, $CDCl_3$):

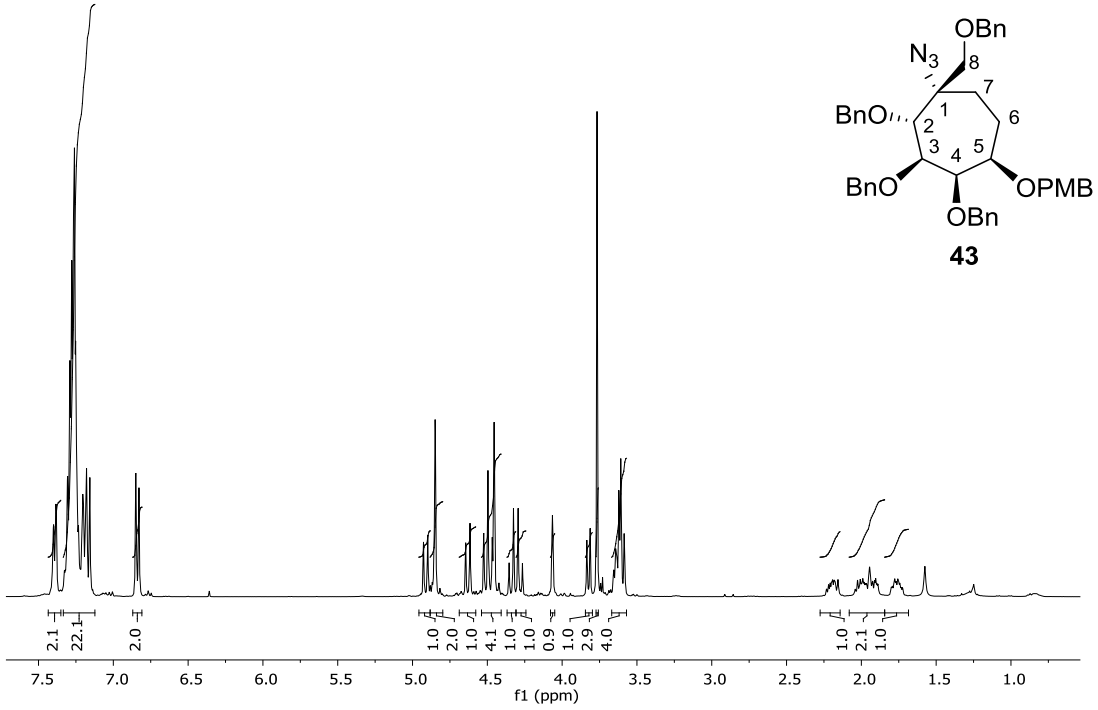


^{13}C-NMR (100 MHz, $CDCl_3$):

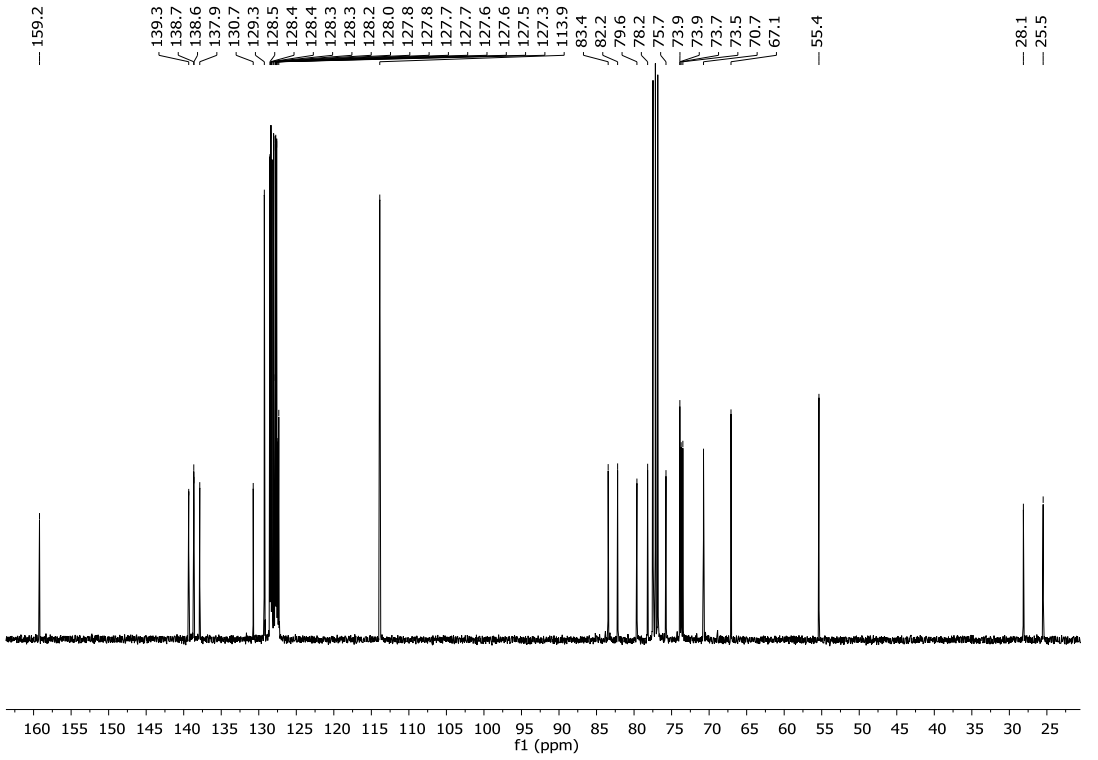


(1*S*,2*R*,3*S*,4*R*,5*R*)-1-azido-2,3,4-tri-*O*-benzyl-1-(benzyloxy)methyl-5-*p*-methoxybenzyl-cycloheptane (43):

^{1}H-NMR (400 MHz, $CDCl_3$):

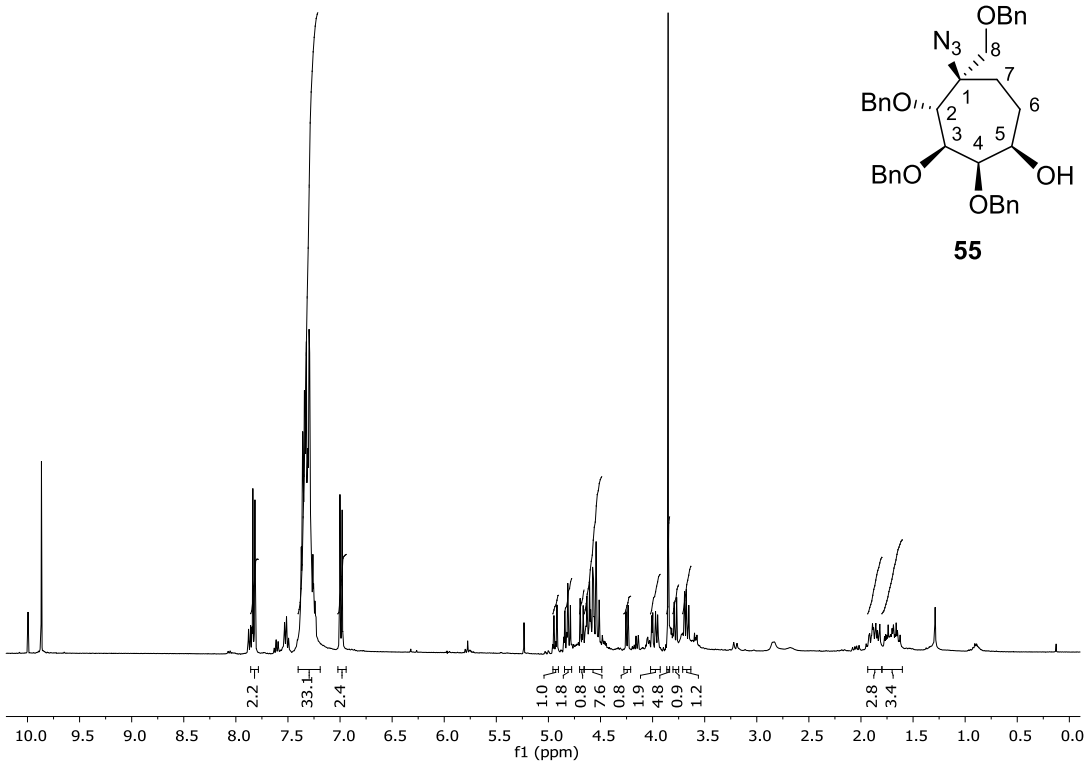


^{13}C-NMR (100 MHz, $CDCl_3$):

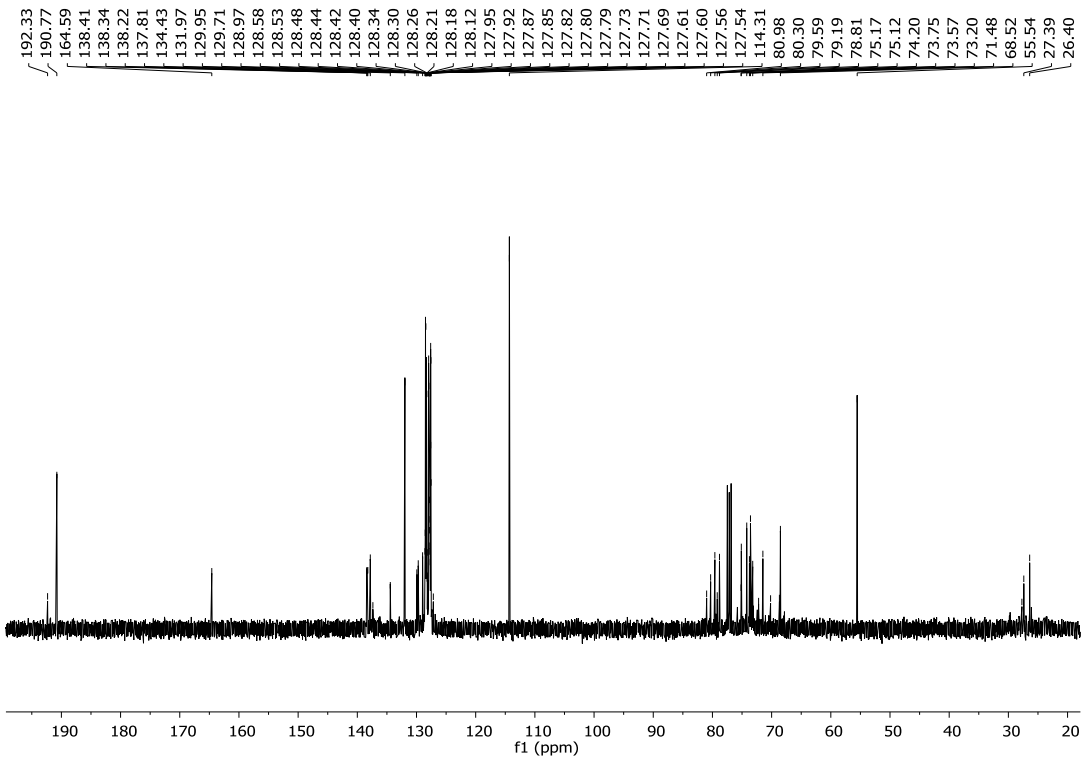


(1*R*,2*R*,3*S*,4*R*,5*R*)-1-azido-2,3,4-tri-*O*-benzyl-1-(benzyloxy)methyl-cycloheptane (55):

^{1}H-NMR (400 MHz, $CDCl_3$):

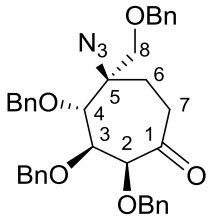


^{13}C-NMR (100 MHz, $CDCl_3$):



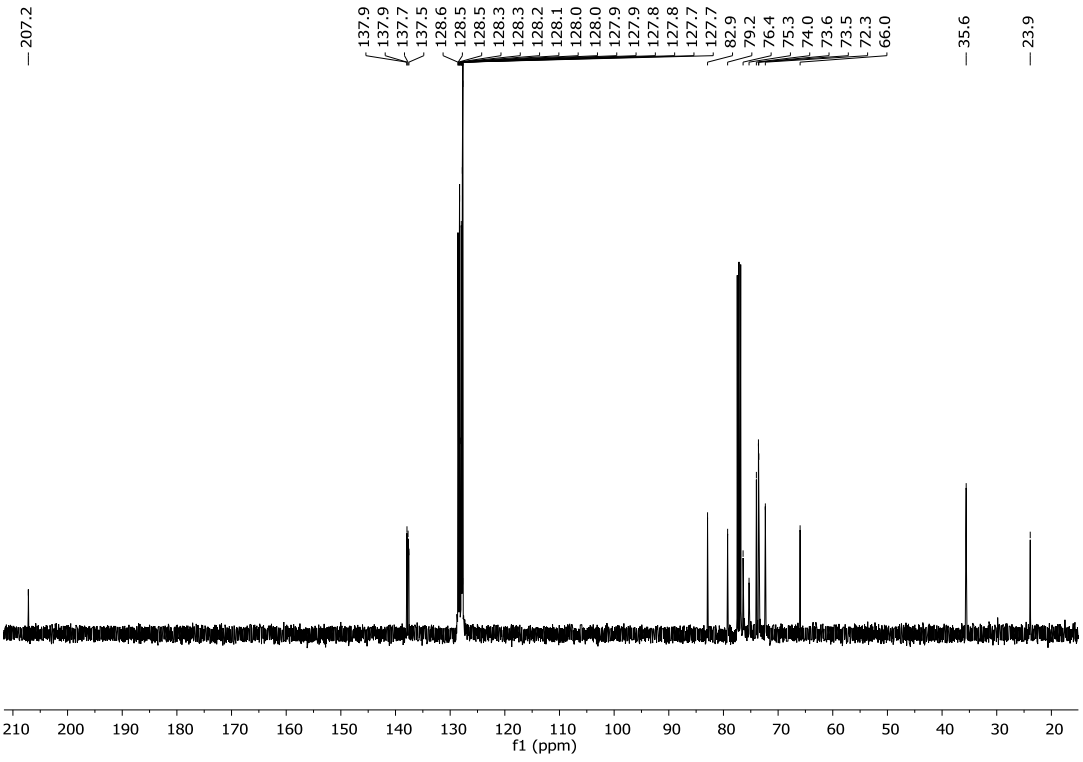
(2*R*,3*S*,4*R*,5*R*)-5-azido-2,3,4-tri-*O*-benzyl-1-(benzyloxy)methyl-cycloheptanone (44):

^{1}H-NMR (400 MHz, $CDCl_3$):



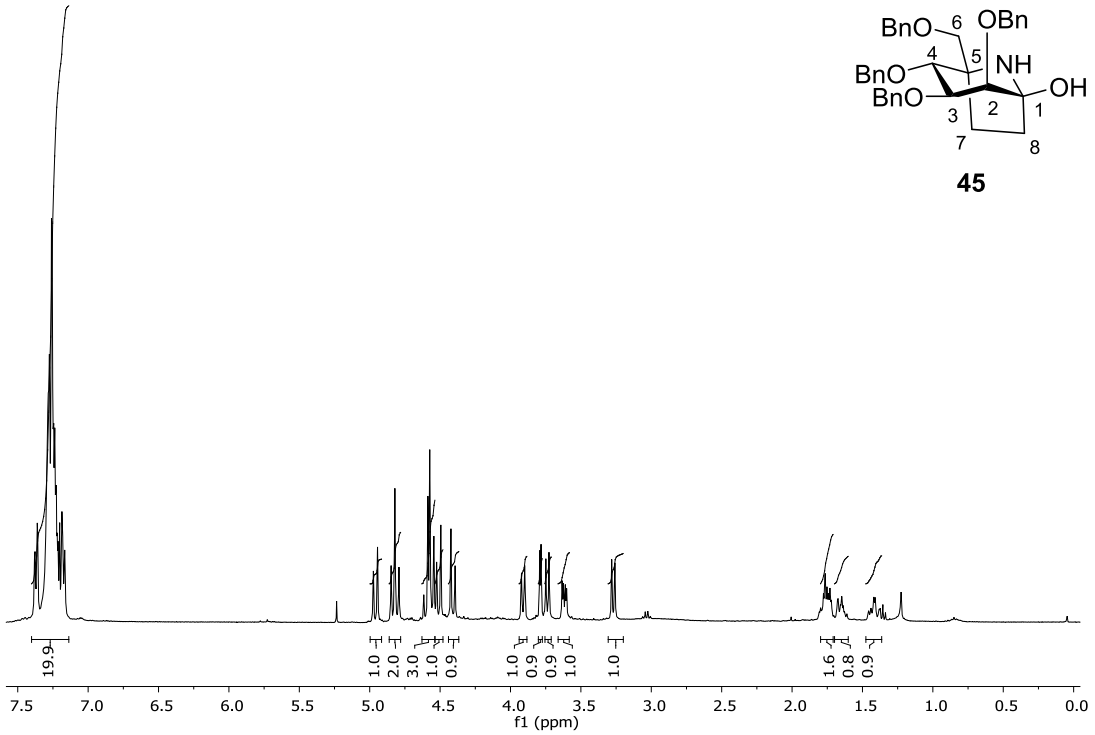
44

^{13}C-NMR (100 MHz, $CDCl_3$):

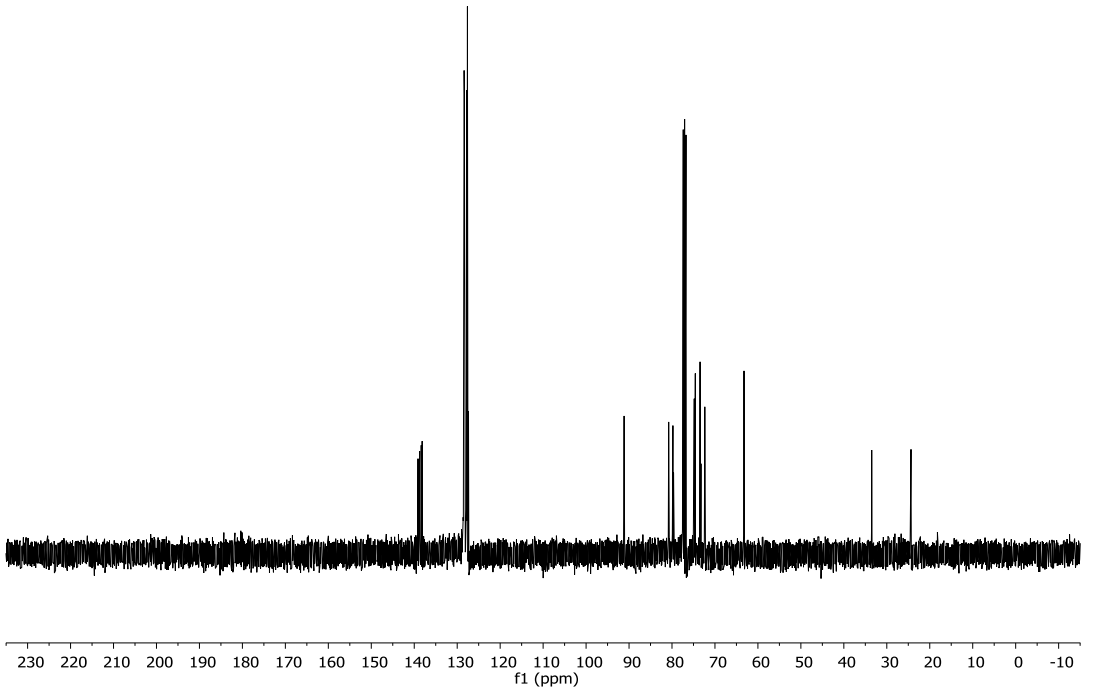


(1S,2S,3S,4R,5R)-2,3,4-tris(benzyloxy)-5-((benzyloxy)methyl)-8-azabicyclo[3.2.1]octan-1-ol (45)

^{1}H-NMR (400 MHz, $CDCl_3$):

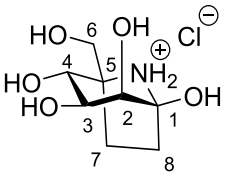


^{13}C-NMR (100 MHz, $CDCl_3$):



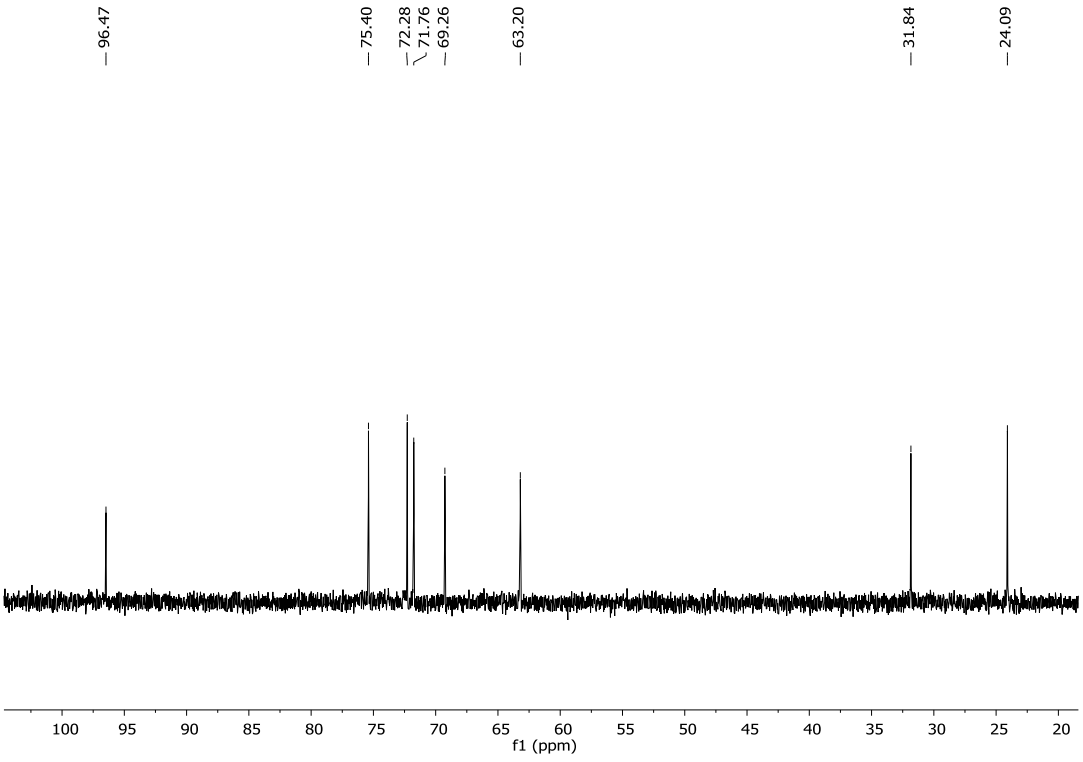
(1S,2S,3S,4R,5R)-5-(hydroxymethyl)-8-azabicyclo[3.2.1]octane-1,2,3,4-tetraol - manno-nojiristegine (2)

^{1}H-NMR (400 MHz, D_2O):



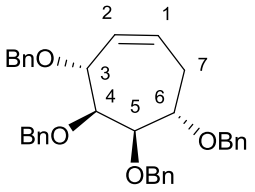
2

^{13}C-NMR (100 MHz, D_2O):



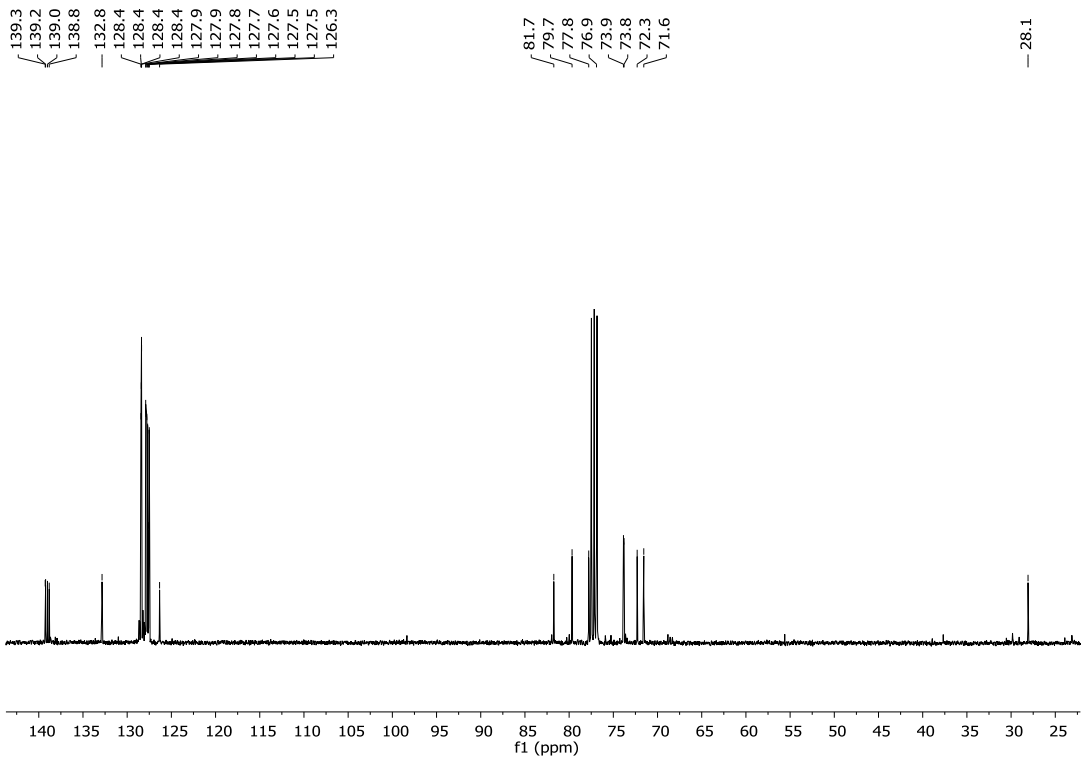
(3*R*,4*S*,5*R*,6*S*)-3,4,5,6-Tetra-*O*-benzyl-cycloheptene (30):

^{1}H-NMR (400 MHz, $CDCl_3$):



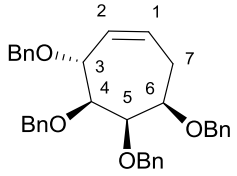
30

^{13}C-NMR (100 MHz, $CDCl_3$):

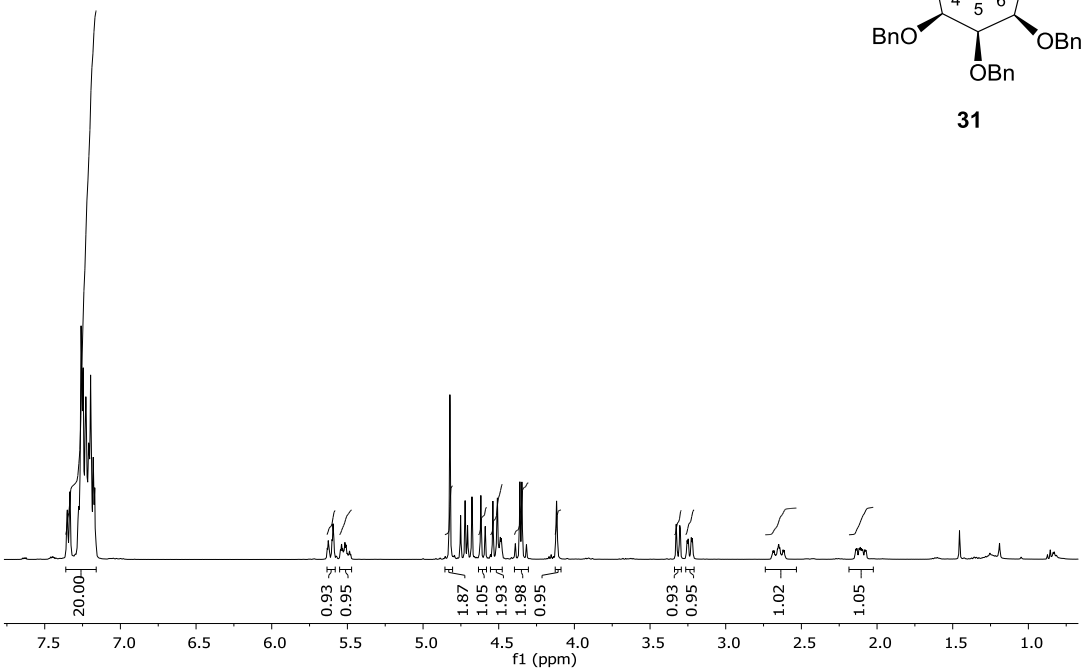


(3*R*,4*S*,5*R*,6*R*)-3,4,5,6-Tetra-*O*-benzyl-cycloheptene (31):

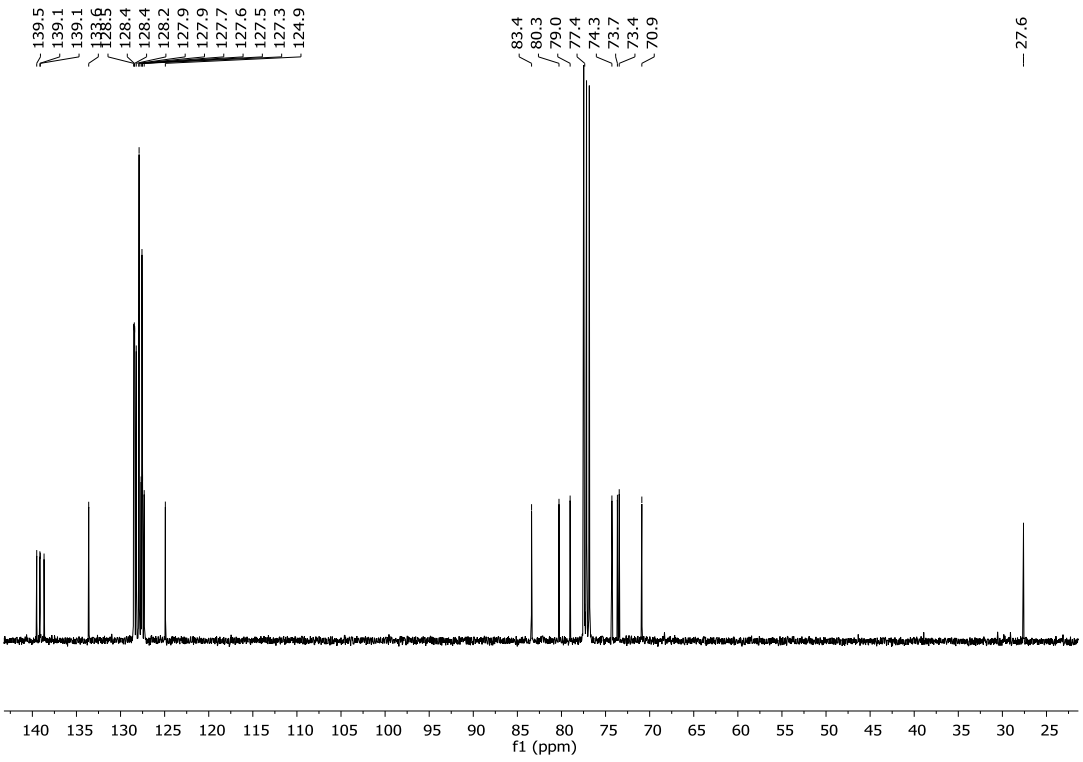
^{1}H-NMR (400 MHz, $CDCl_3$):



31

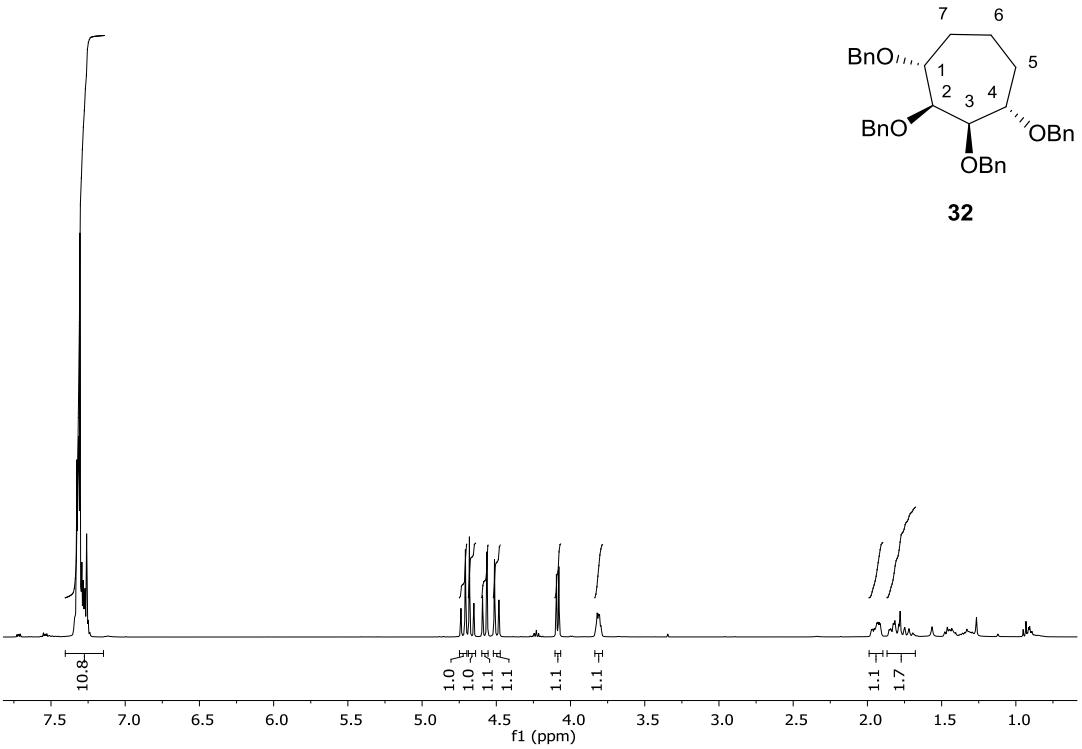


^{13}C-NMR (100 MHz, $CDCl_3$):

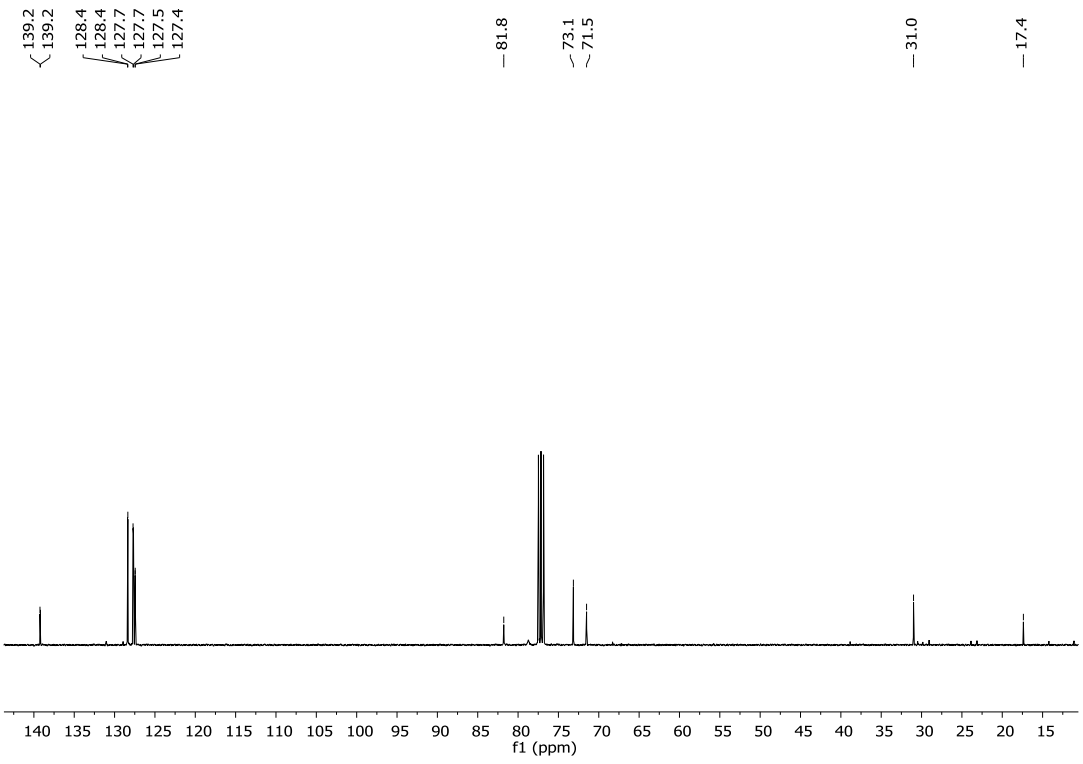


(1*R*,2*S*,3*R*,4*S*)-1,2,3,4-Tetra-*O*-benzl-cycloheptane (32):

^{1}H-NMR (400 MHz, $CDCl_3$):

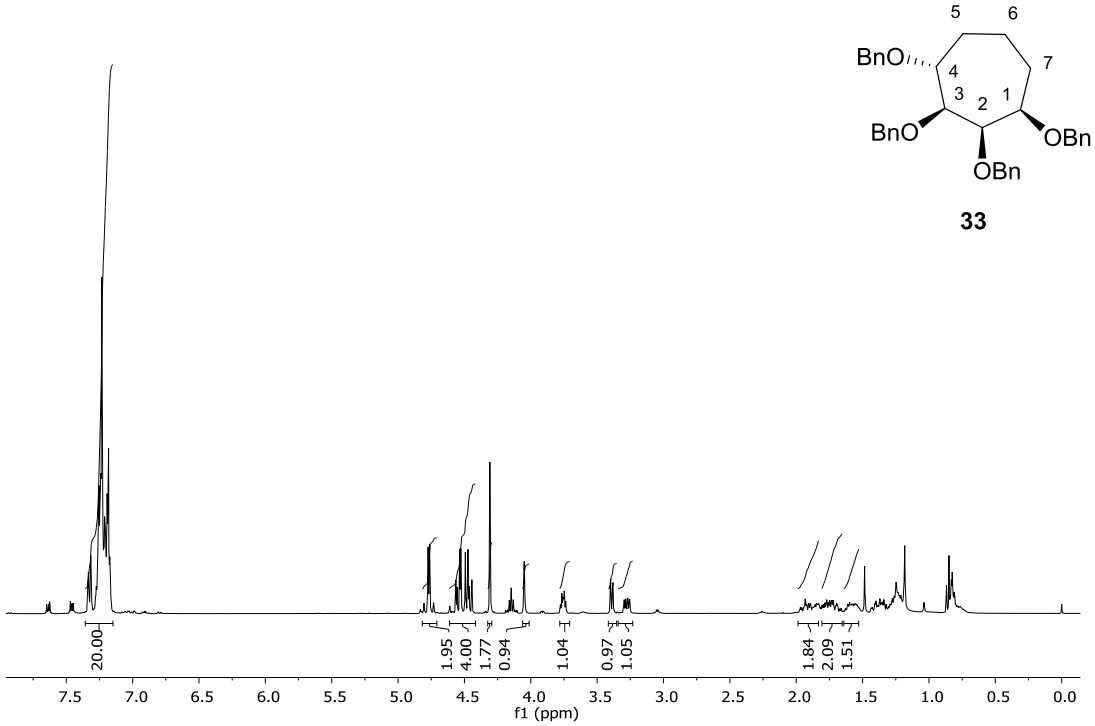


^{13}C-NMR (100 MHz, $CDCl_3$):



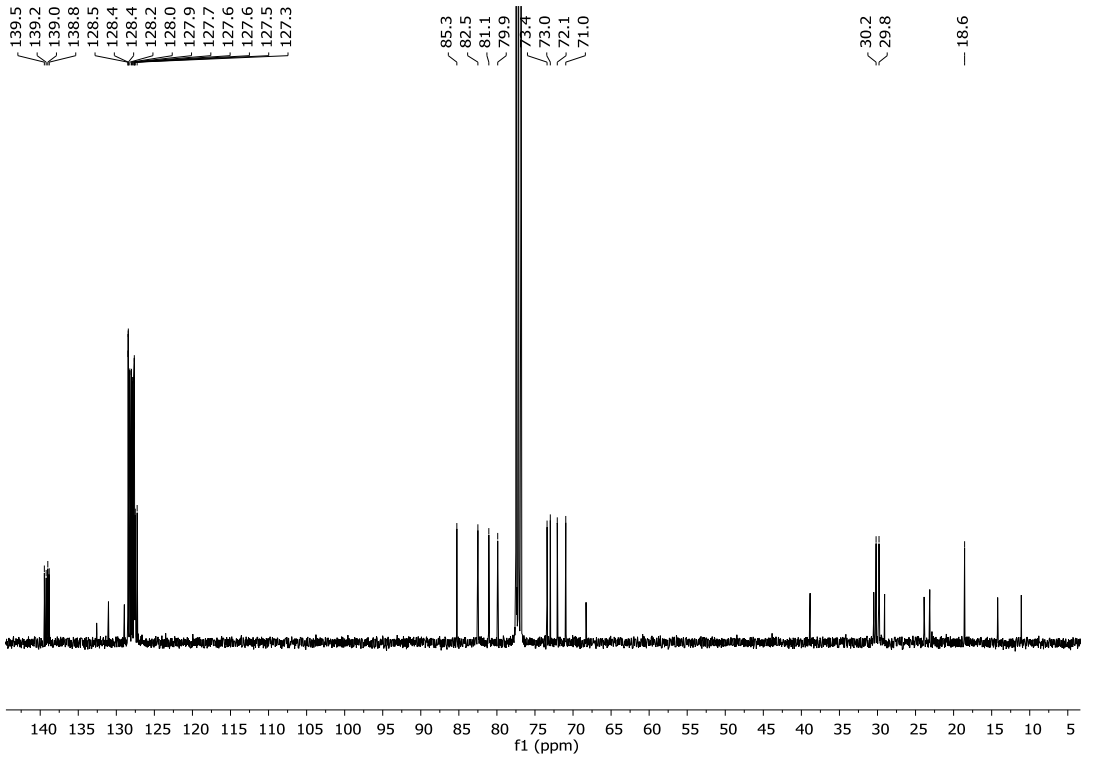
(1*R*,2*S*,3*R*,4*R*)-1,2,3,4-Tetra-*O*-benzyl-cycloheptane (33):

^{1}H-NMR (400 MHz, $CDCl_3$):



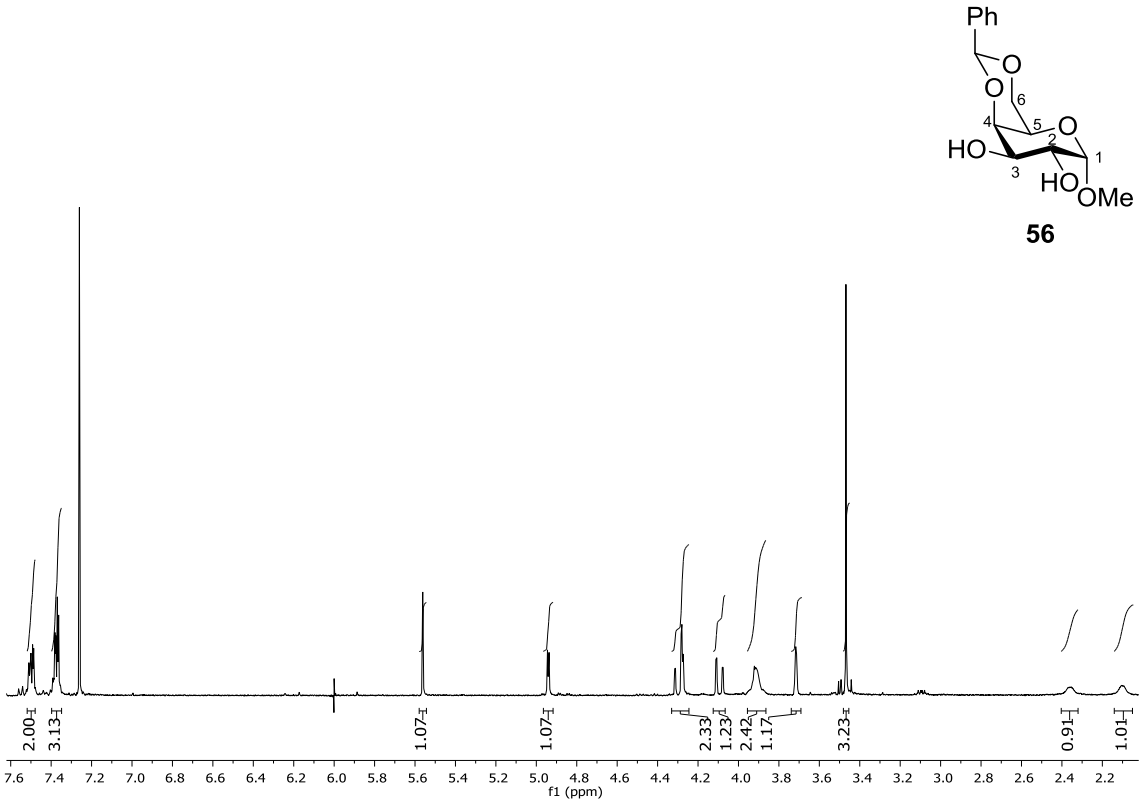
33

^{13}C-NMR (100 MHz, $CDCl_3$):

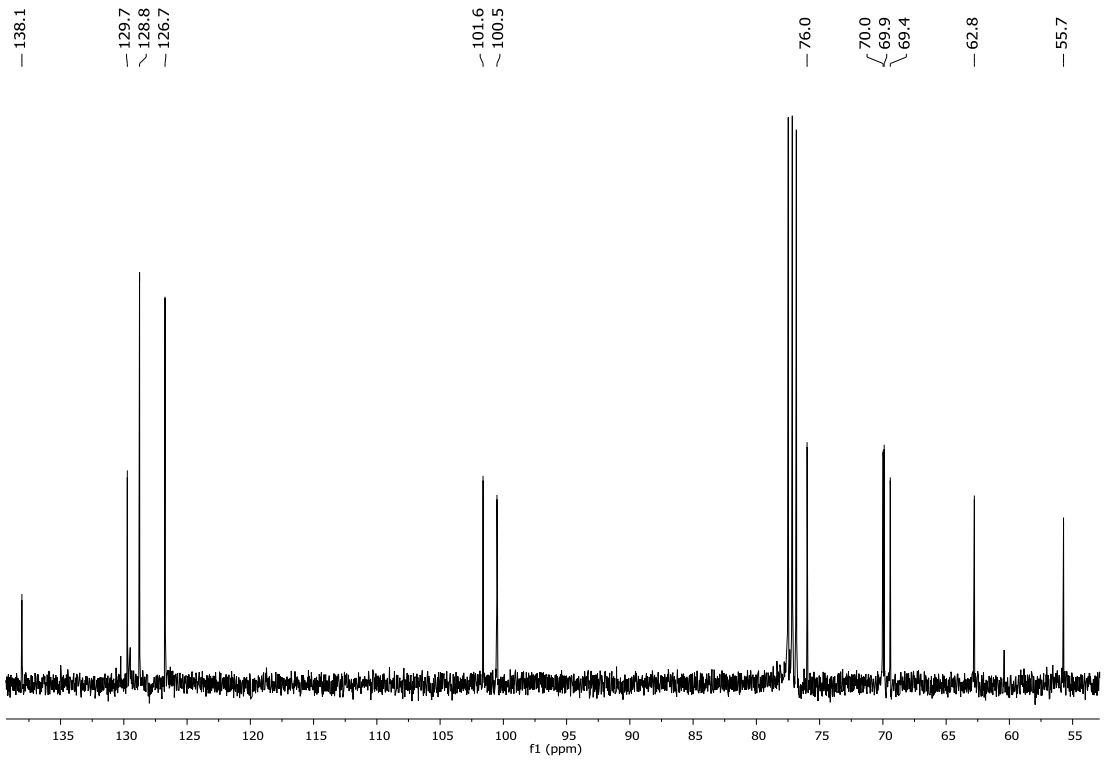


Methyl 4,6-*O*-Benzylidene-*α*- D-galactopyranoside (56):

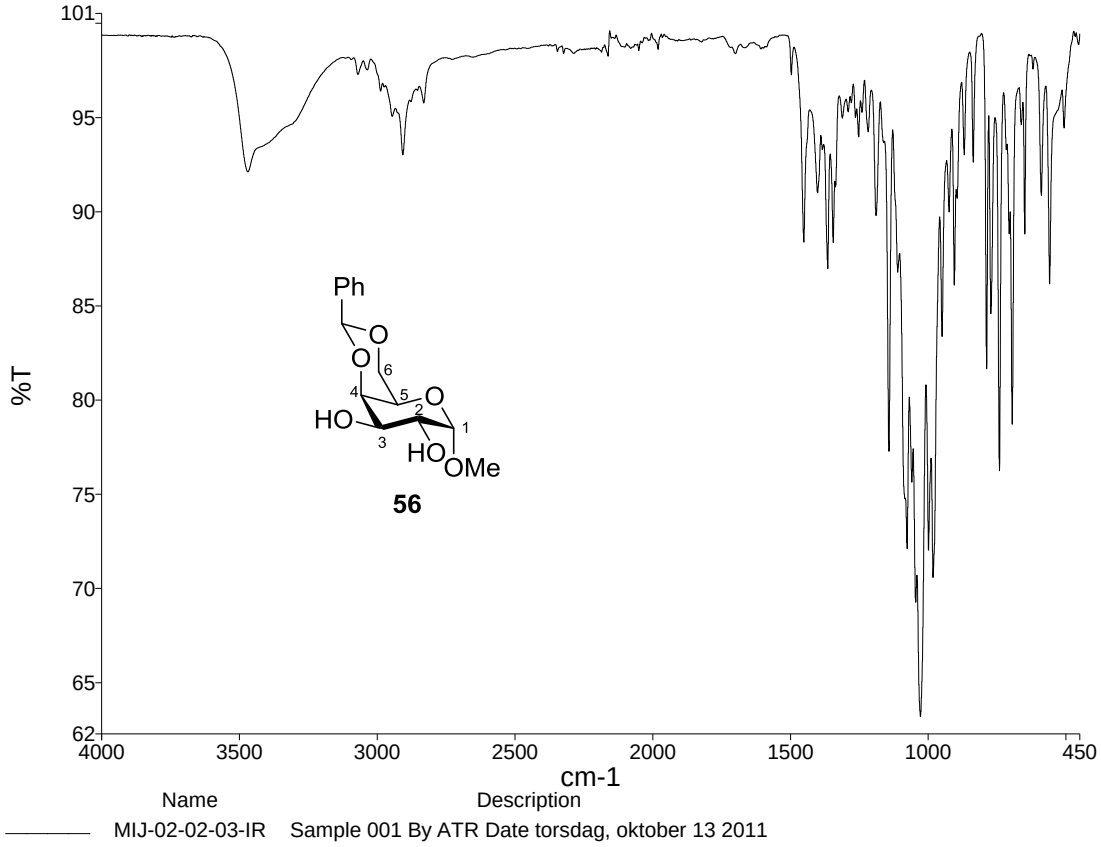
^{1}H-NMR (400 MHz, $CDCl_3$):



^{13}C-NMR (100 MHz, $CDCl_3$):

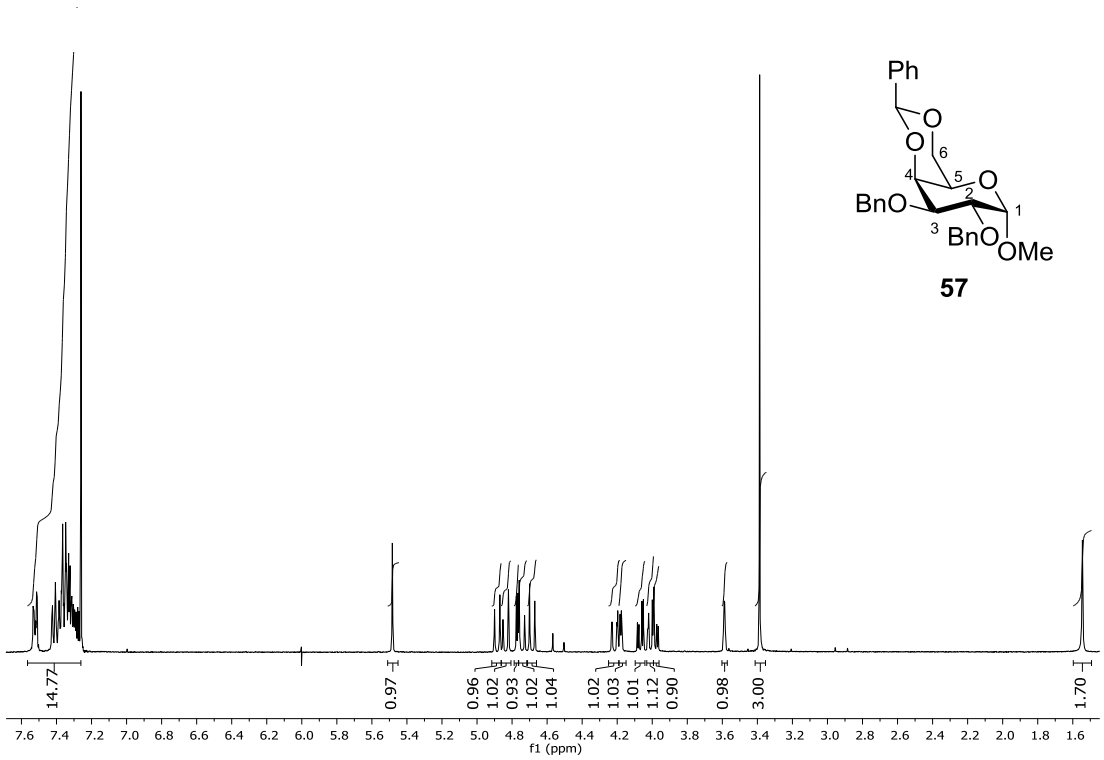


IR (neat):

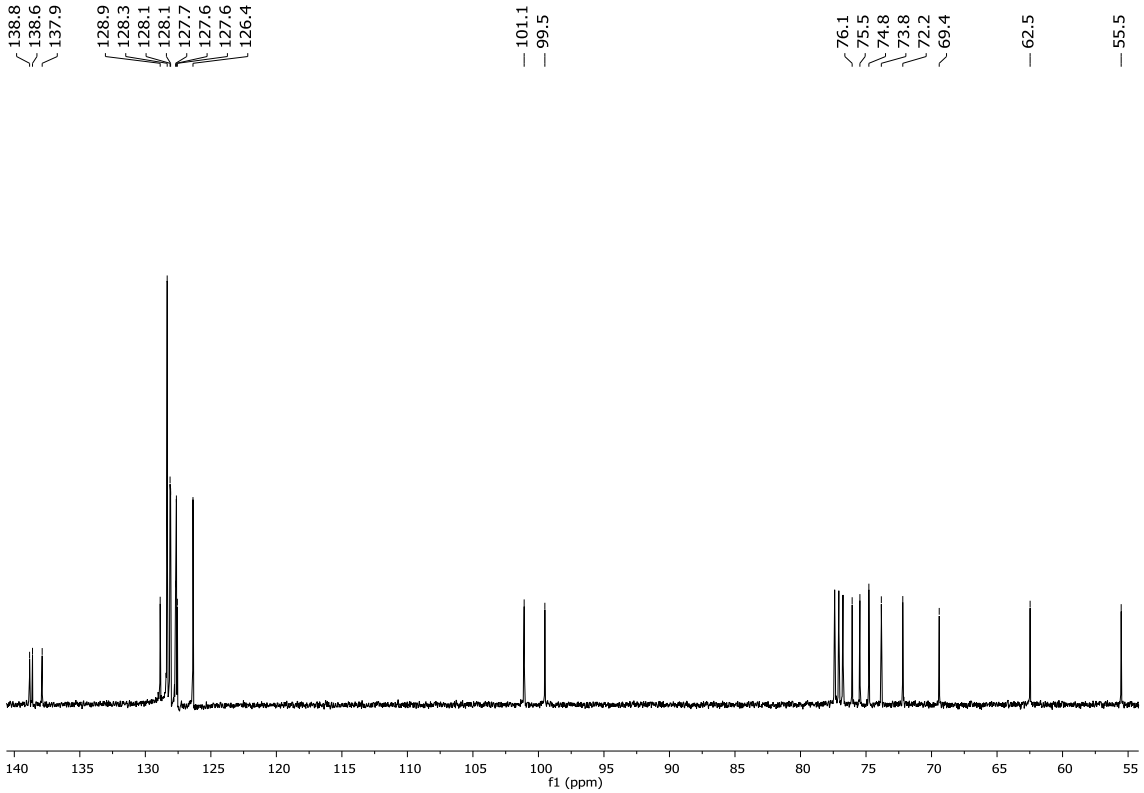

101
95
90
85
80
75
70
65
62
%T
4000
3500
3000
2500
2000
1500
1000
450
cm-1
Ph
O
O
6
4
5
O
2
1
HO
3
HO
OMe
56
Name
Description
MIJ-02-02-03-IR
Sample 001 By ATR Date torsdag, oktober 13 2011

Methyl 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-*α*- D-galactopyranoside (57):

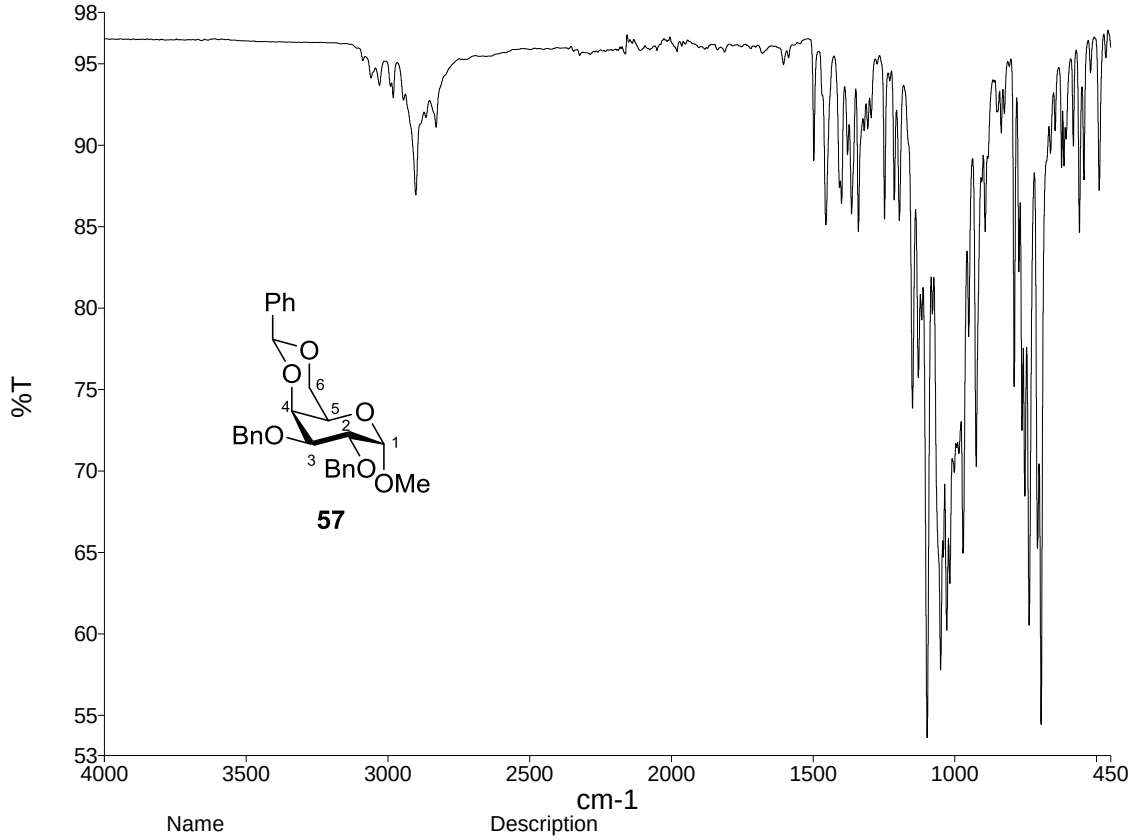
^{1}H-NMR (400 MHz, $CDCl_3$):



^{13}C-NMR (100 MHz, $CDCl_3$):

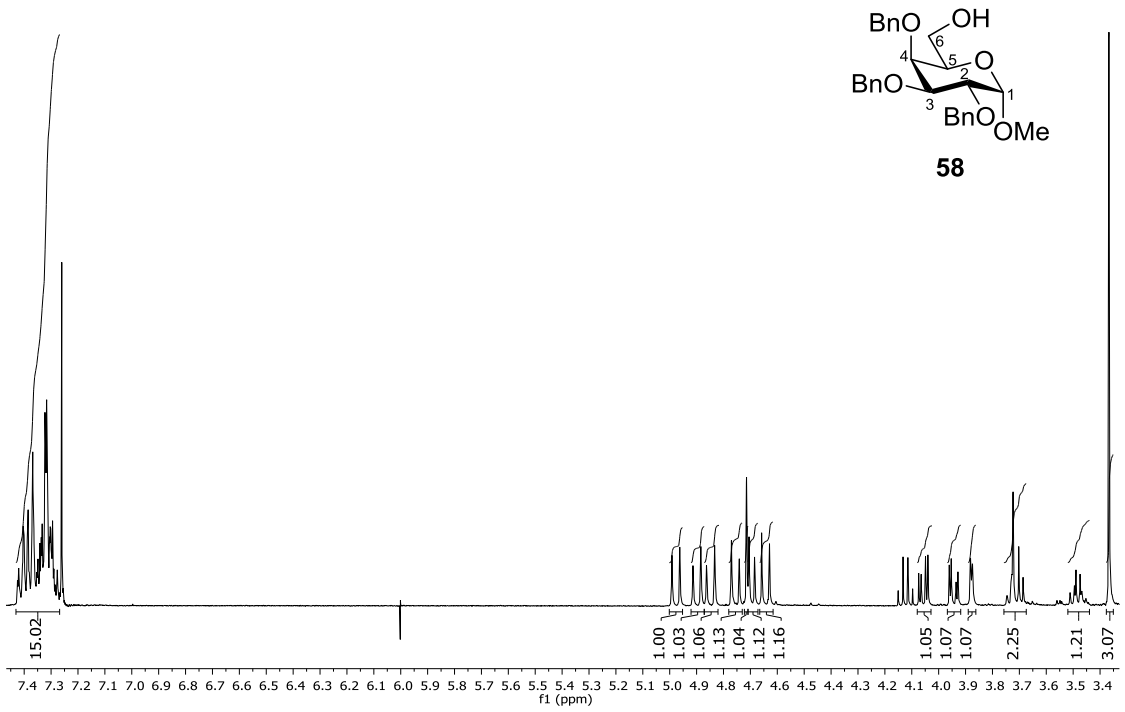


IR (neat):

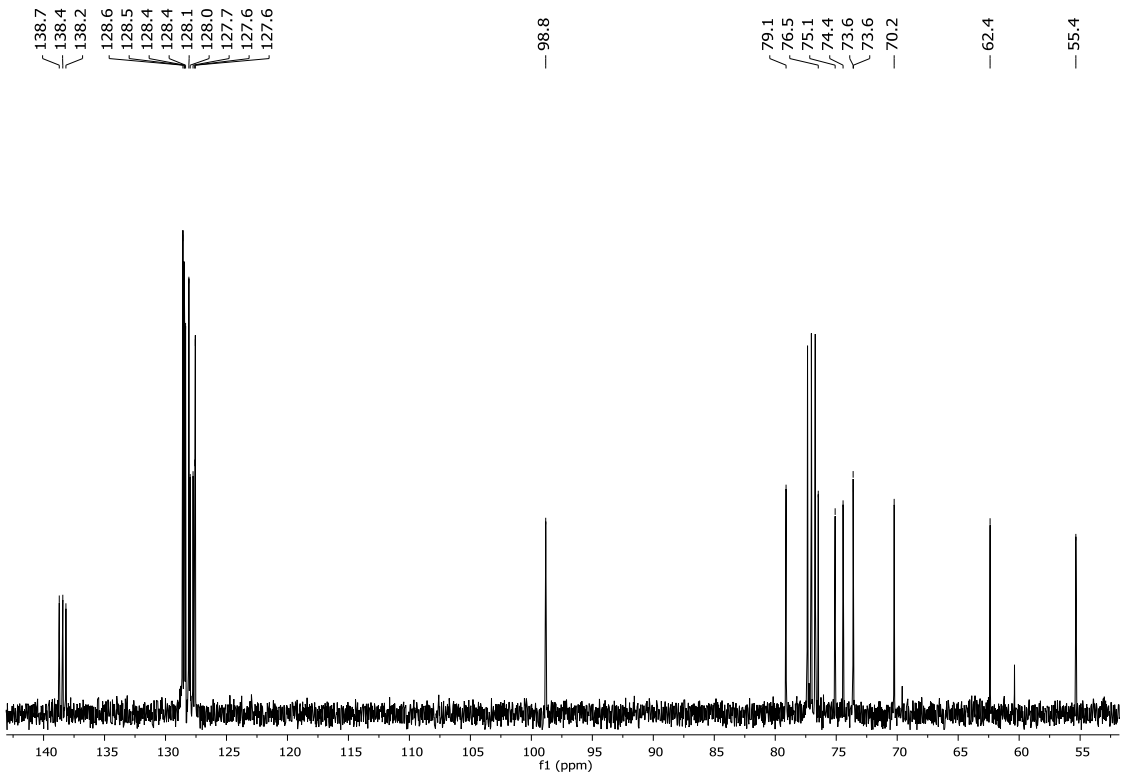

98
95
90
85
80
75
70
65
60
55
53
%T
4000
3500
3000
2500
2000
1500
1000
450
cm-1
Ph
O
O
6
4
5
O
2
1
BnO
3
BnO
OMe
57
Name
Description
MIJ-02-03-07-IR
Sample 004 By ATR Date torsdag, oktober 13 2011

Methyl 2,3,4-tri-*O*-benzyl-6-hydroxy-α- D-galactopyranoside (58):

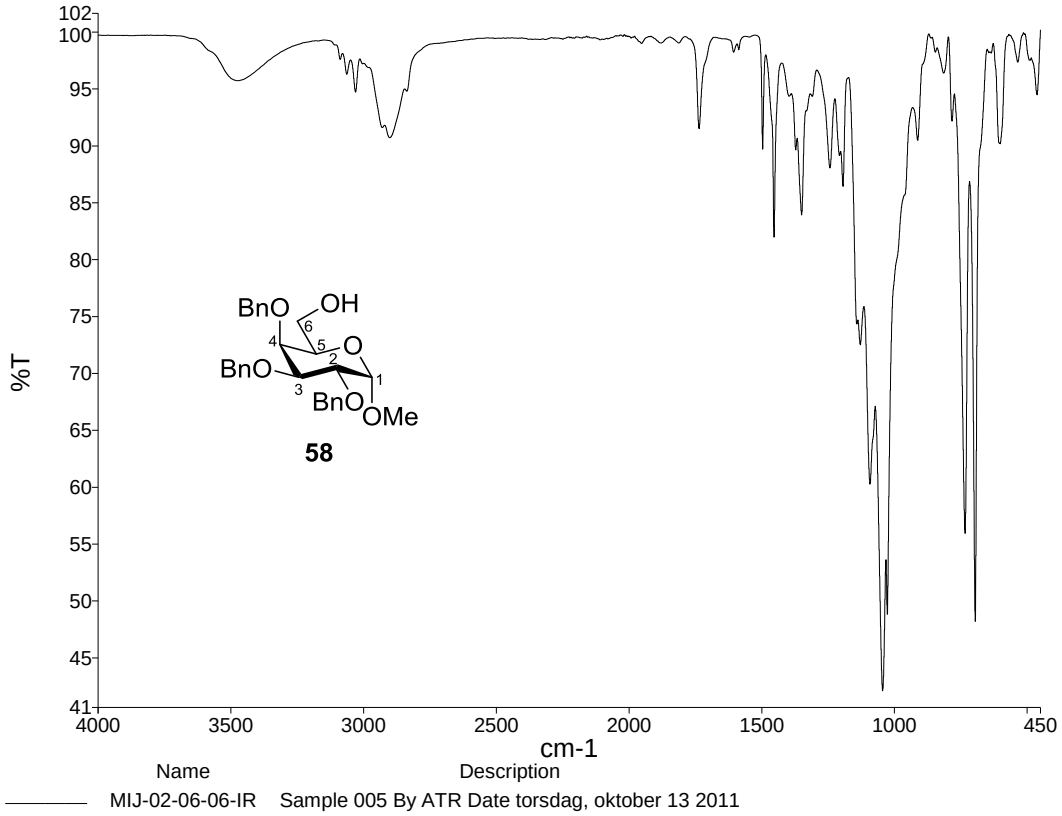
^{1}H-NMR (400 MHz, $CDCl_3$):



^{13}C-NMR (100 MHz, $CDCl_3$):

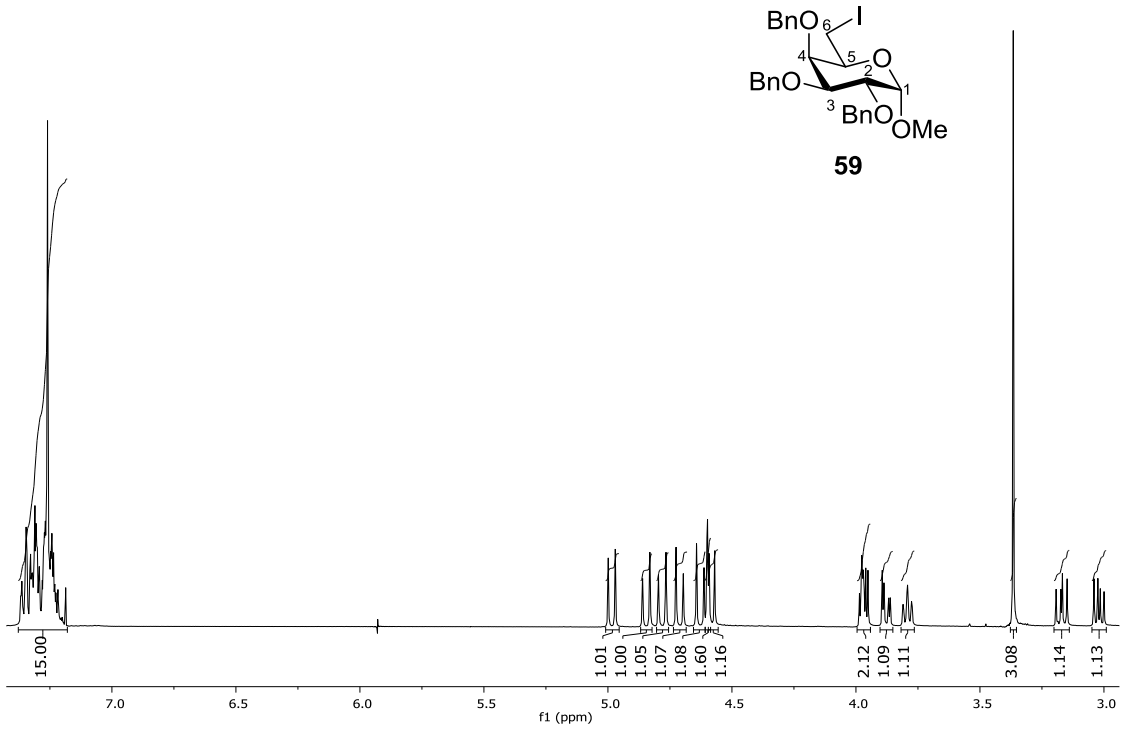


IR (neat):

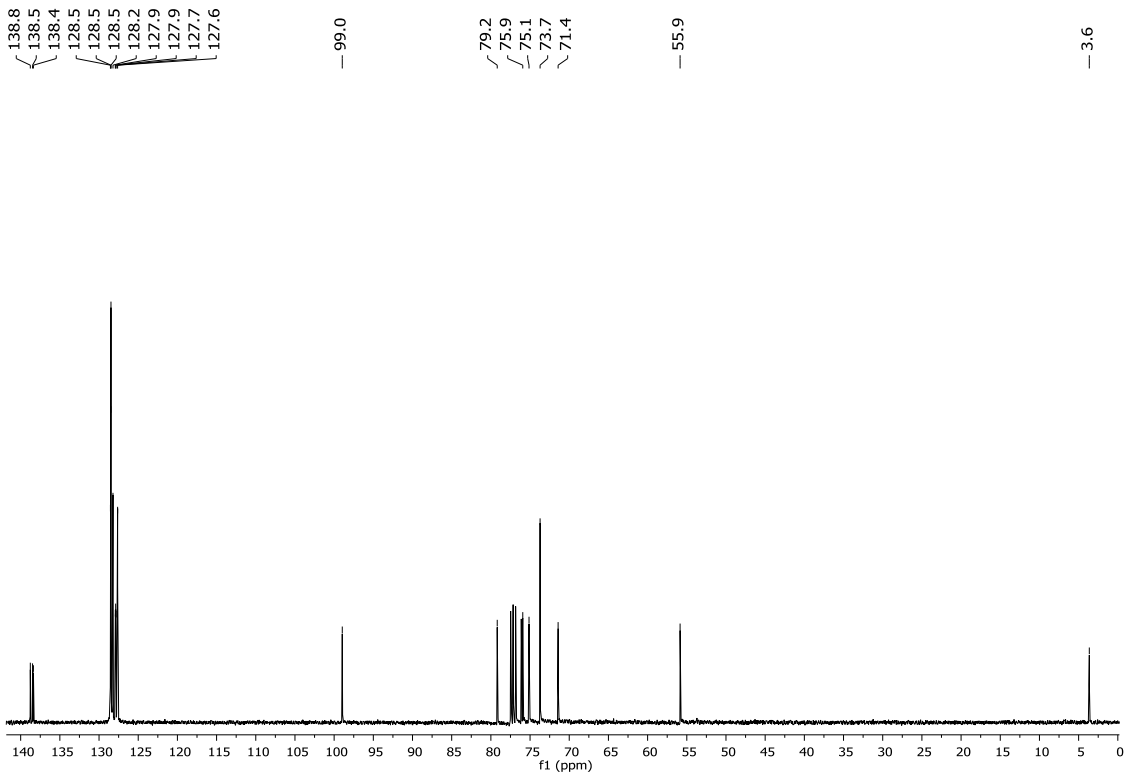

%T
cm-1
BnO
OH
O
BnO
BnO
OMe
58
Name
Description
MIJ-02-06-06-IR Sample 005 By ATR Date torsdag, oktober 13 2011

Methyl 2,3,4-tri-*O*-benzyl-6-deoxy-6-iodo-α- D-galactopyranoside (59):

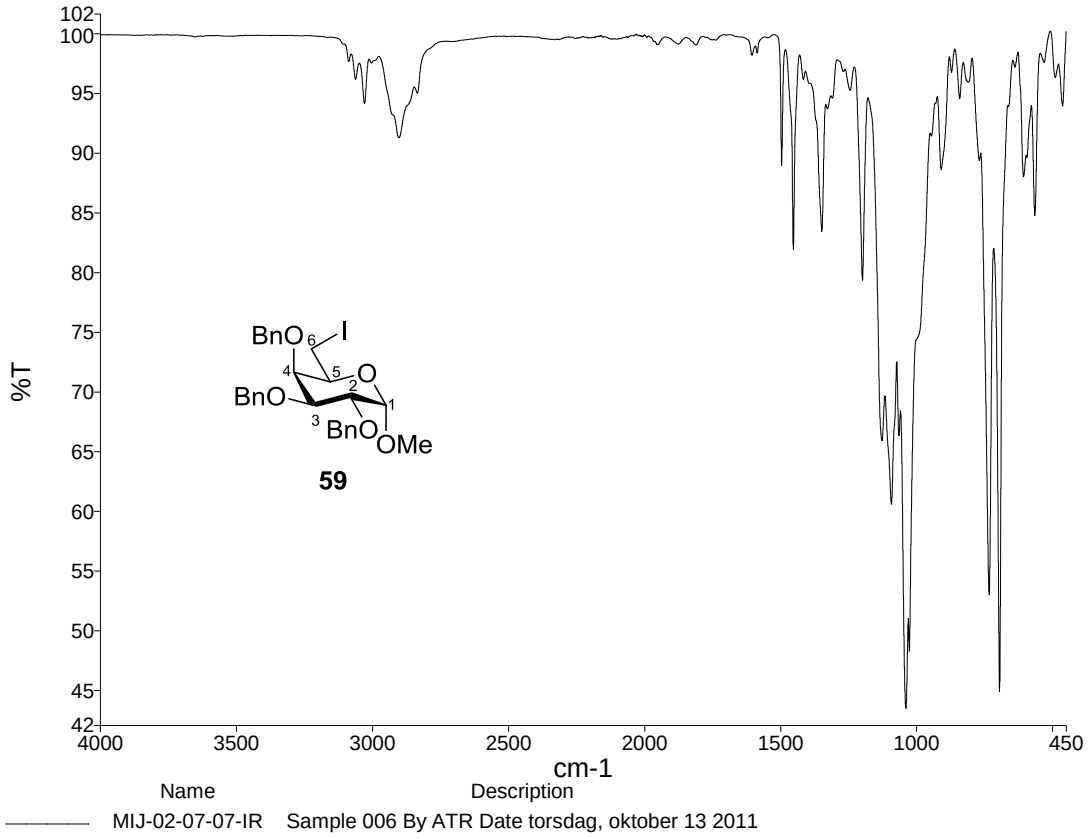
^{1}H-NMR (400 MHz, $CDCl_3$):



^{13}C-NMR (100 MHz, $CDCl_3$):

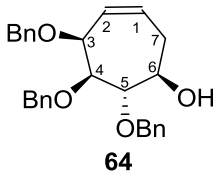


IR (neat):


%T
cm-1
BnO
I
O
OMe
59
Name
Description
MIJ-02-07-07-IR Sample 006 By ATR Date torsdag, oktober 13 2011

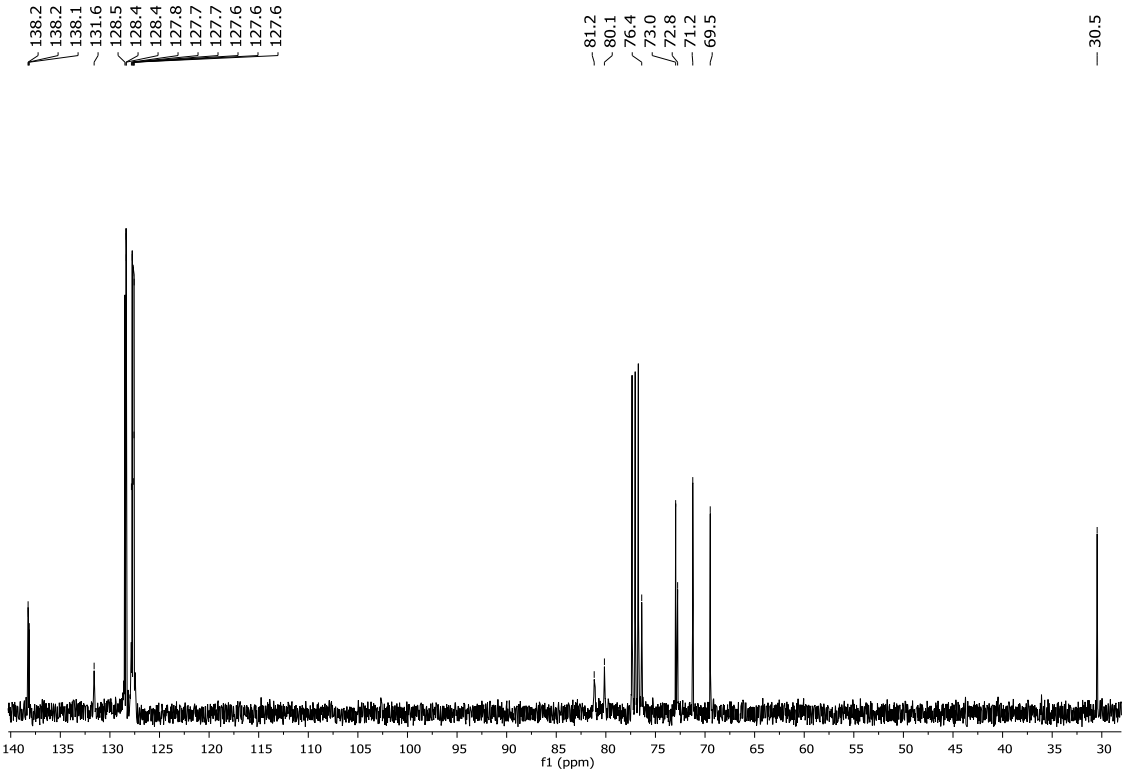
(3*S*,4*S*, 5*S*, 6*R*)-3,4,5-Tri-*O*-benzyl-6-hydroxyl-cycloheptene (64):

^{1}H-NMR (400 MHz, $CDCl_3$):

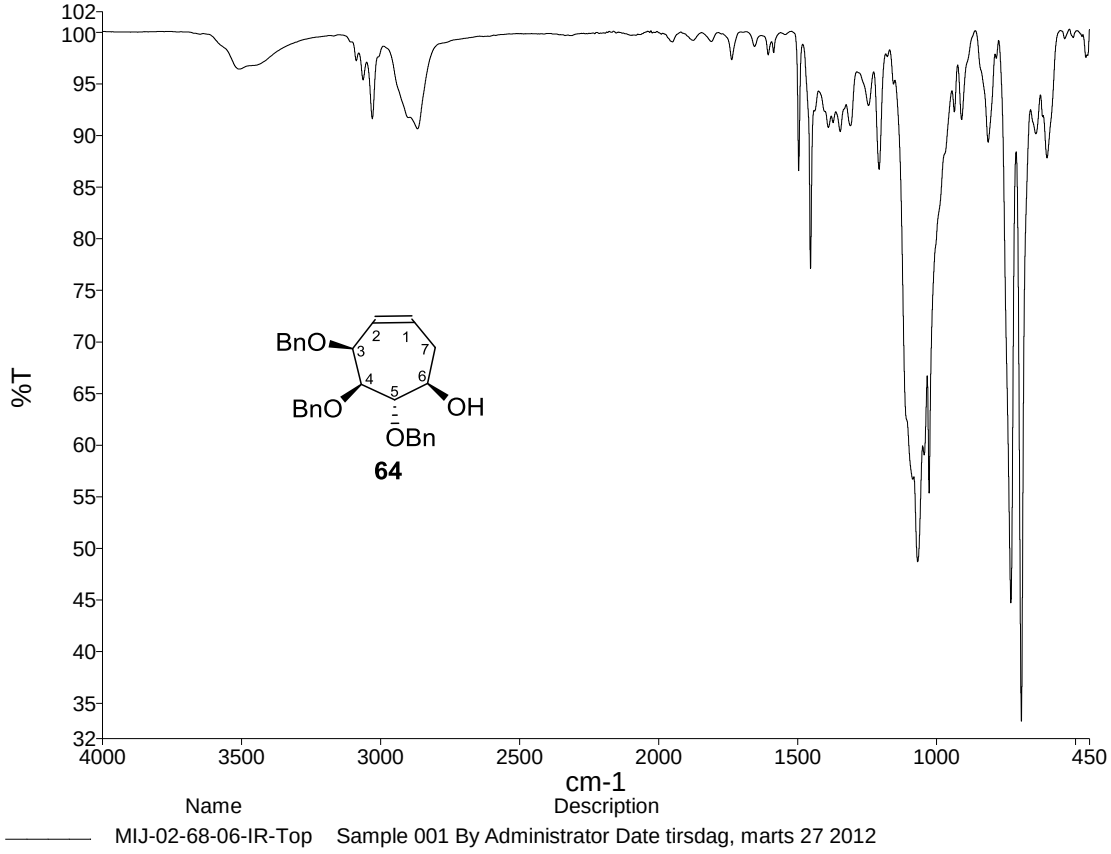


64

^{13}C-NMR (100 MHz, $CDCl_3$):

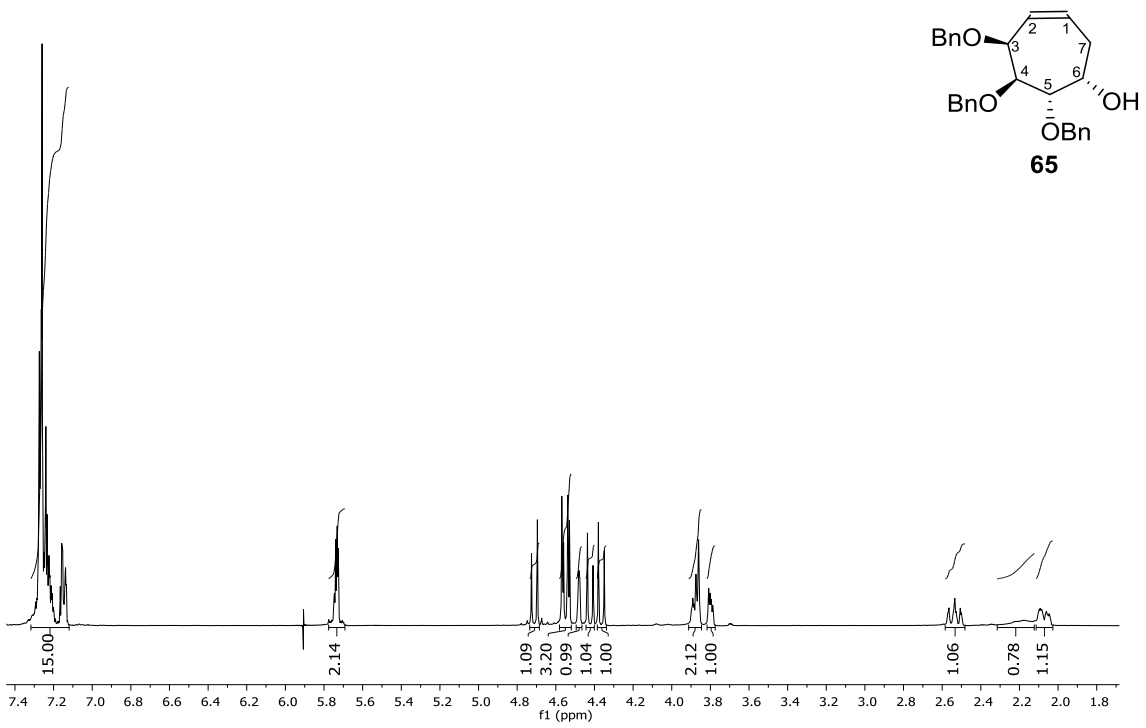


IR (neat):

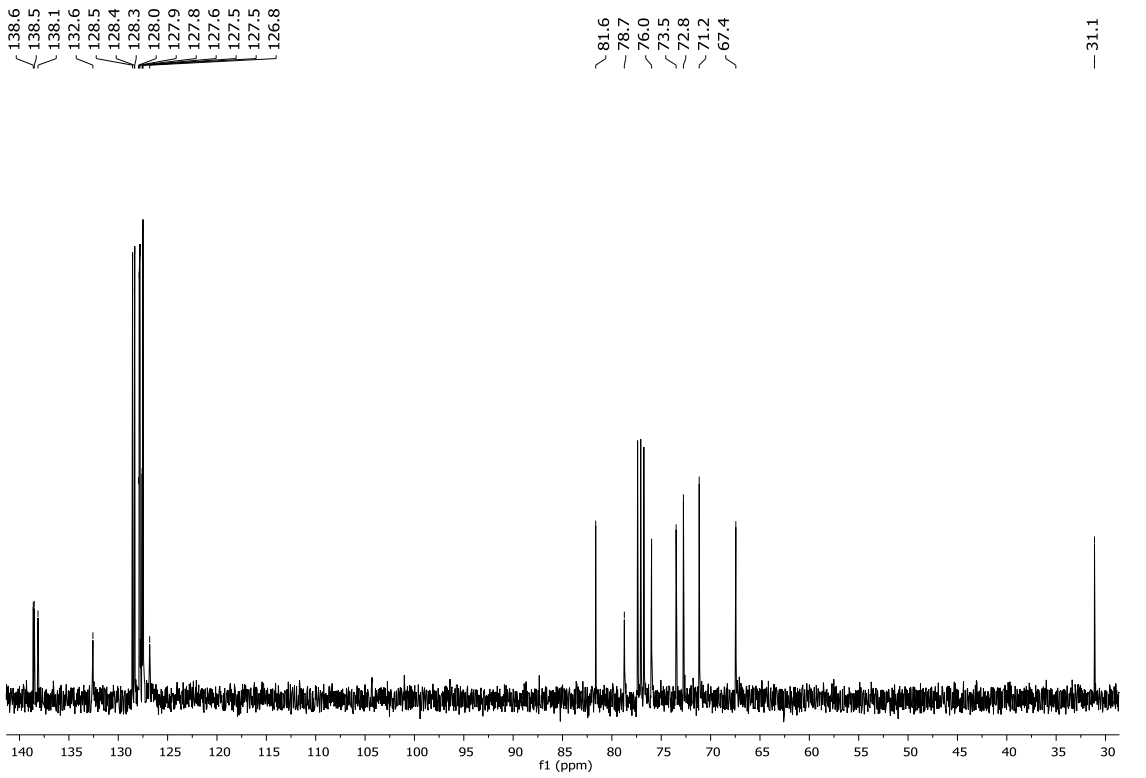

%T
cm-1
4000
3500
3000
2500
2000
1500
1000
450
BnO
OH
BnO
OBn
64
Name
Description
MIJ-02-68-06-IR-Top
Sample 001 By Administrator Date tirsdag, marts 27 2012

(3*S*,4*S*, 5*S*, 6*S*)-3,4,5-Tri-*O*-benzyl-6-hydroxy-cycloheptene (65):

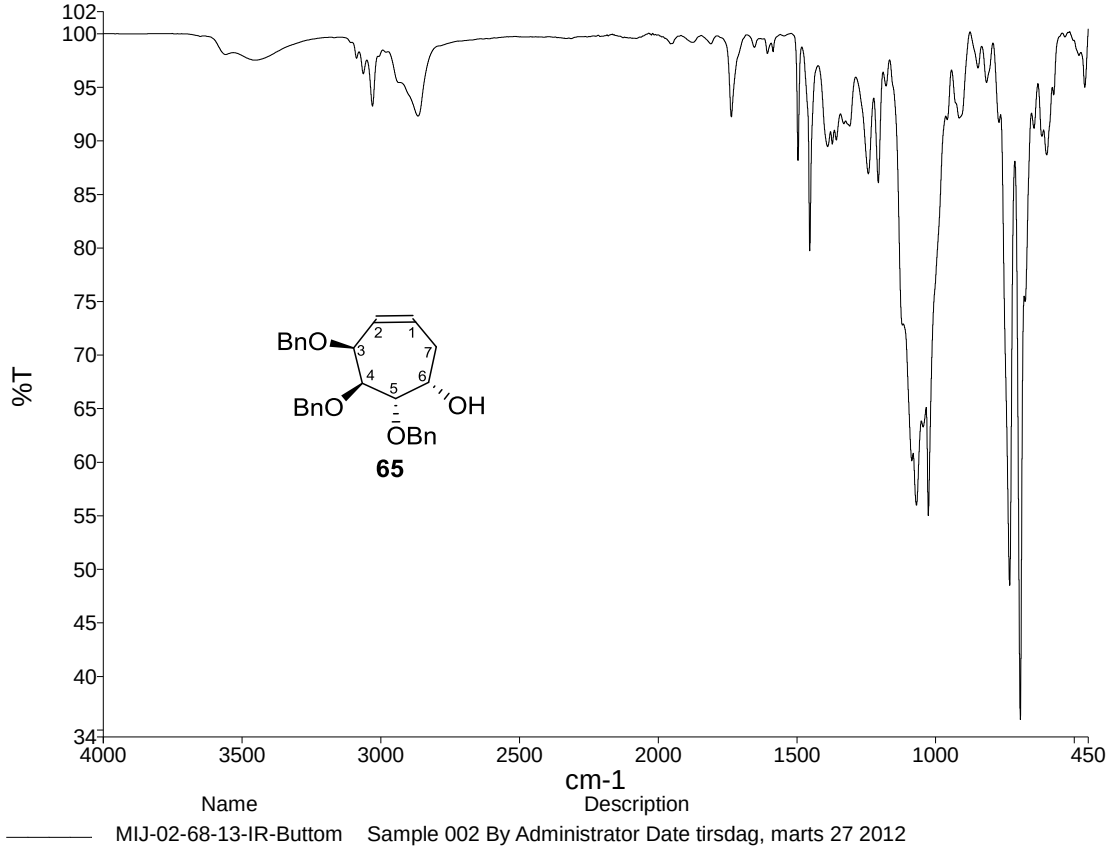
^{1}H-NMR (400 MHz, $CDCl_3$):



^{13}C-NMR (100 MHz, $CDCl_3$):

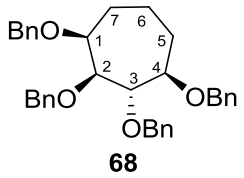


IR (neat):


%T
cm-1
4000
3500
3000
2500
2000
1500
1000
450
BnO
BnO
OH
OBn
65
Name
Description
MIJ-02-68-13-IR-Buttom Sample 002 By Administrator Date tirsdag, marts 27 2012

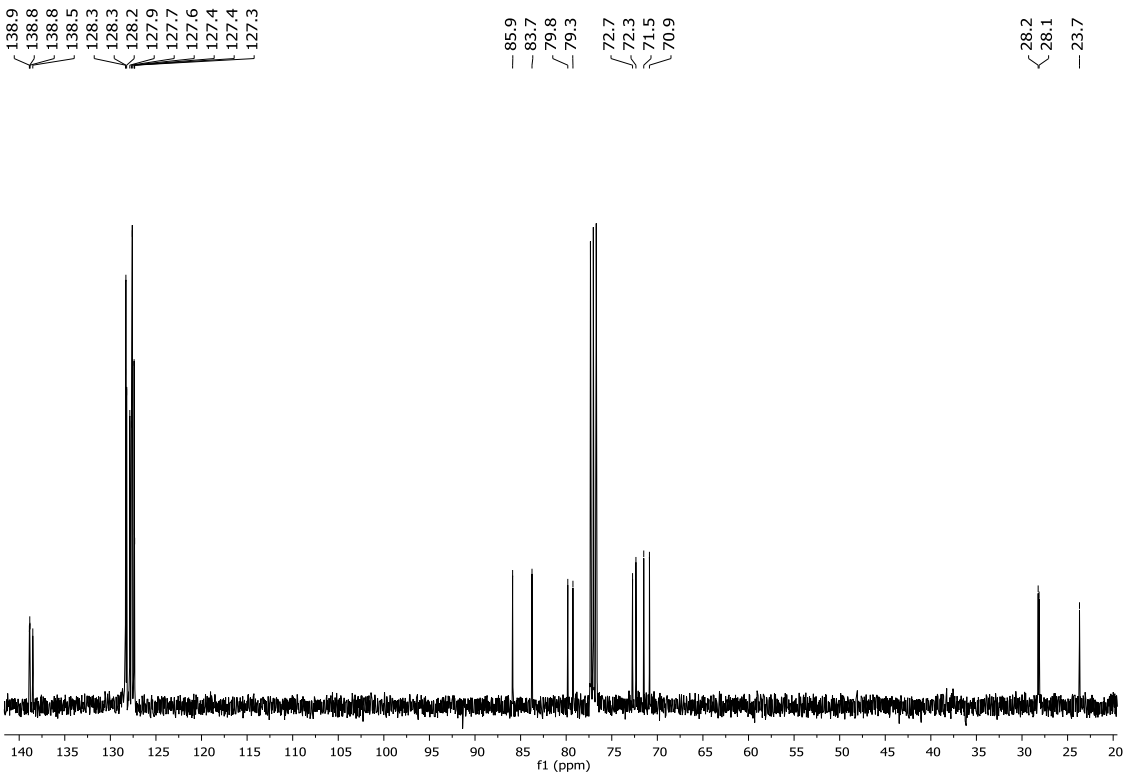
(1*S*, 2*S*, 3*S*, 4*R*)-1,2,3,5-Tetra-*O*-benzyl-cycloheptane (68):

^{1}H-NMR (400 MHz, $CDCl_3$):

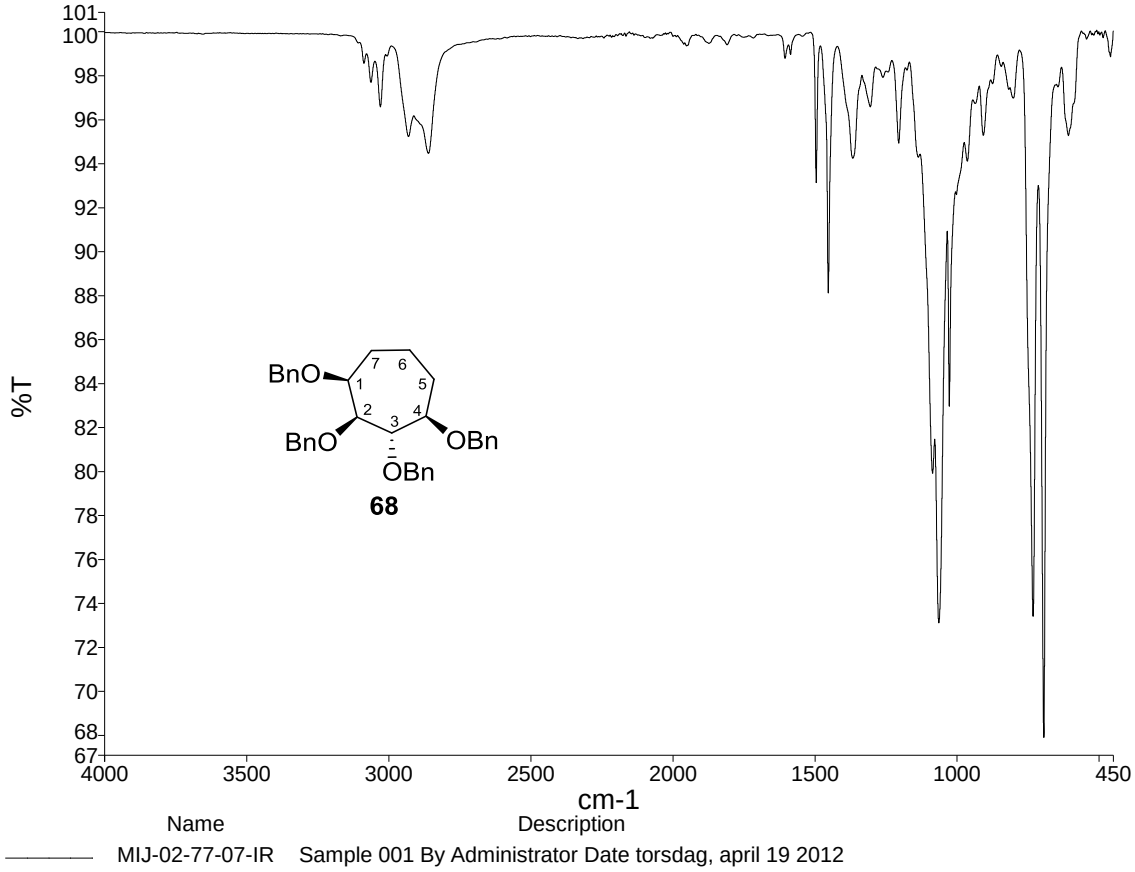


68

^{13}C-NMR (100 MHz, $CDCl_3$):

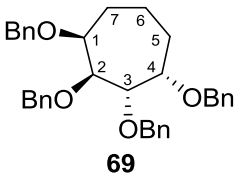


IR (neat):


%T
cm-1
BnO
OBn
BnO
OBn
68
Name
Description
MIJ-02-77-07-IR Sample 001 By Administrator Date torsdag, april 19 2012

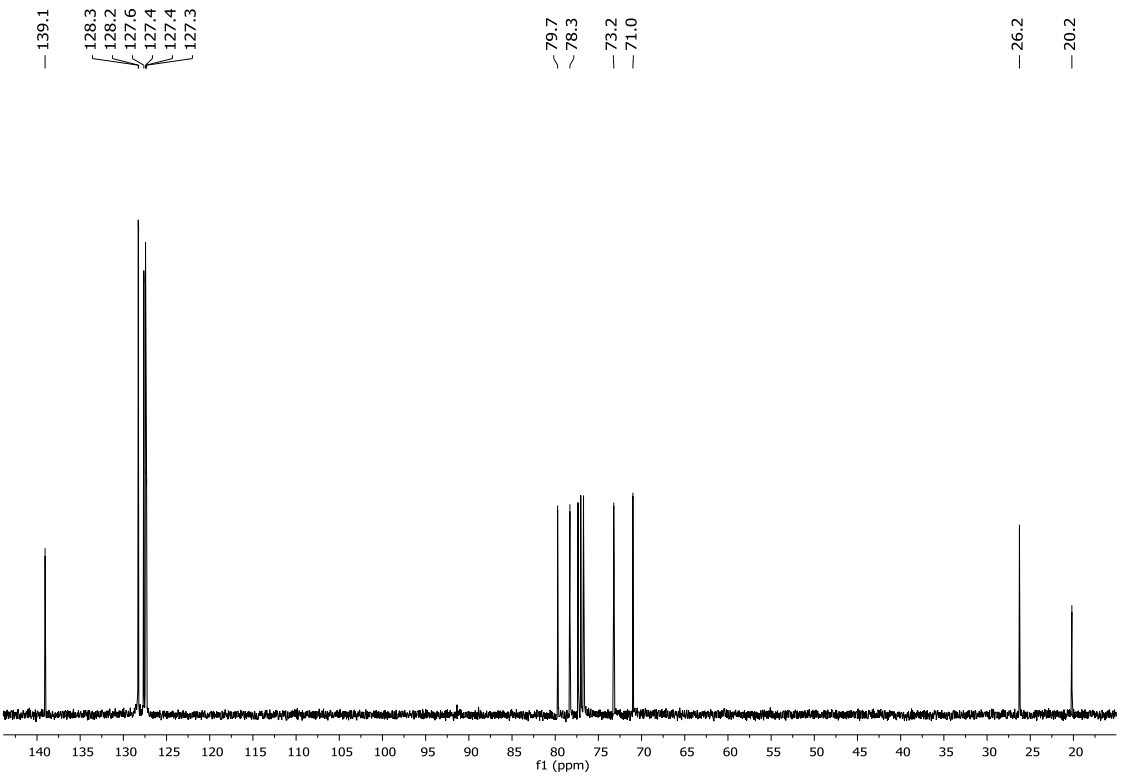
(1*S*, 2*S*, 3*S*, 4*S*)-1,2,3,4-Tetra-*O*-benzyl-cycloheptane (69):

^{1}H-NMR (400 MHz, $CDCl_3$):

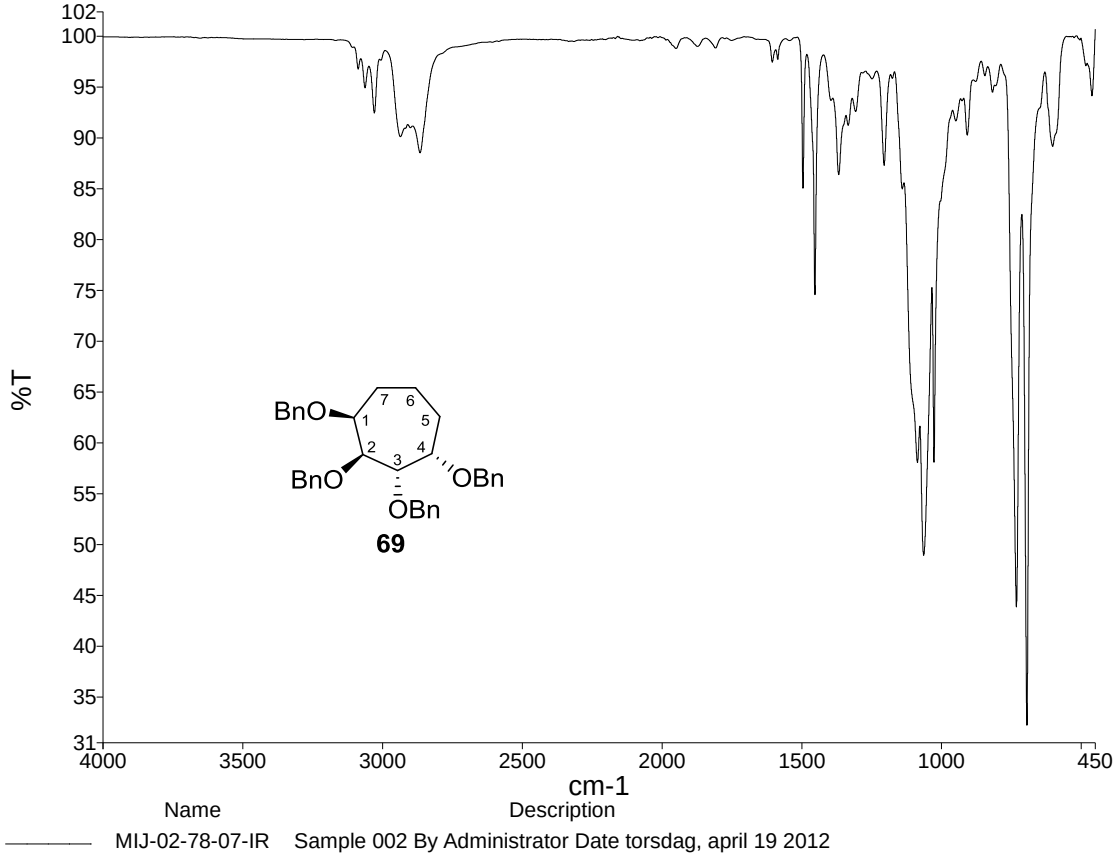


69

^{13}C-NMR (100 MHz, $CDCl_3$):



IR (neat):


%T
cm-1
4000
3500
3000
2500
2000
1500
1000
450
BnO
BnO
OBn
OBn
69
Name
Description
MIJ-02-78-07-IR Sample 002 By Administrator Date torsdag, april 19 2012